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AUGUST 17, 1942
VOLUME 31, No. 17

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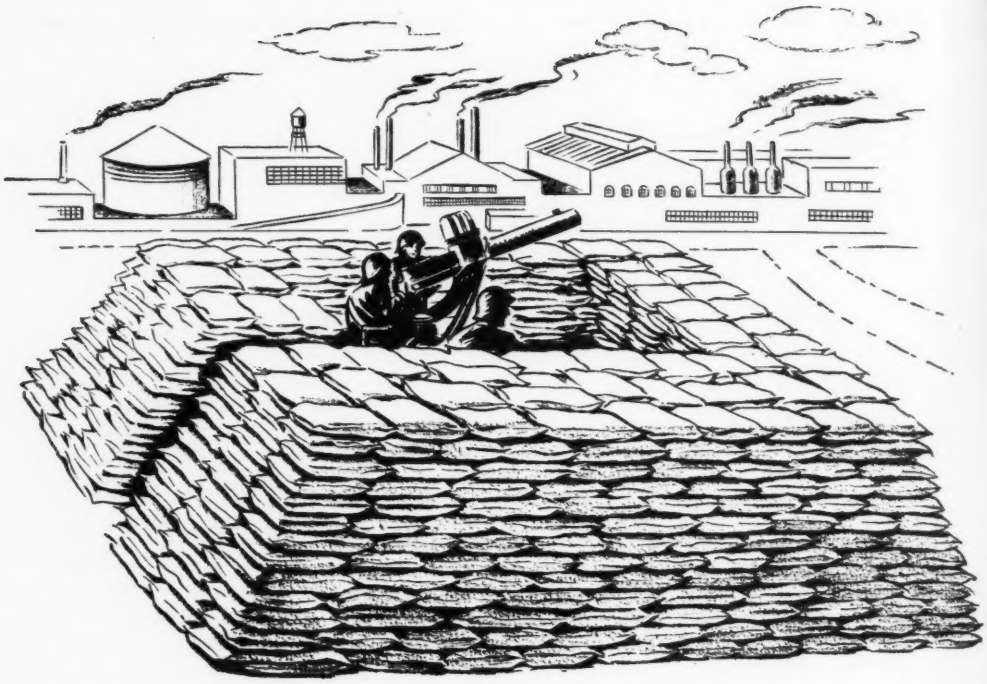

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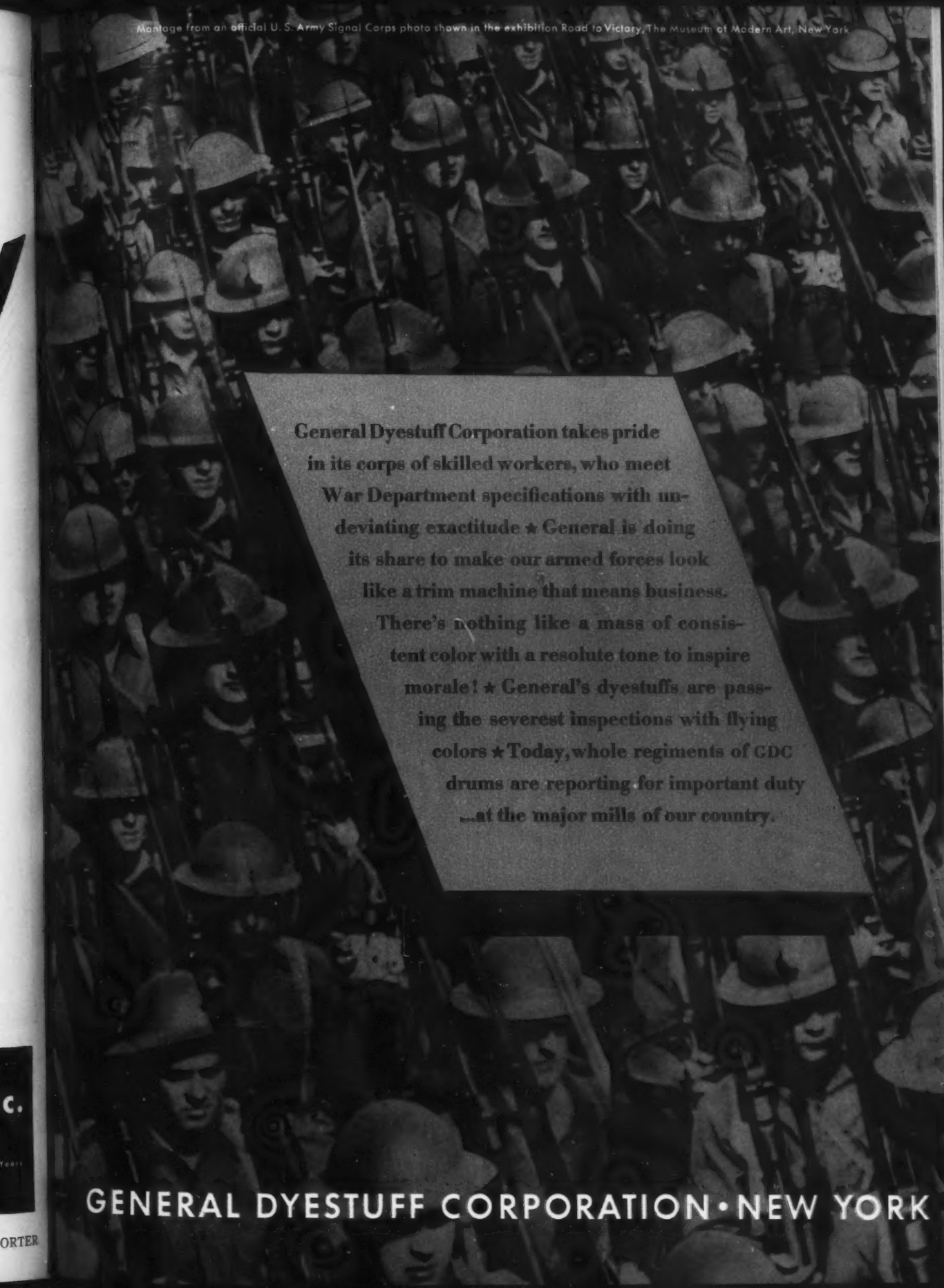
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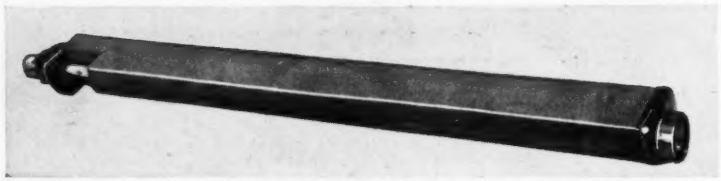

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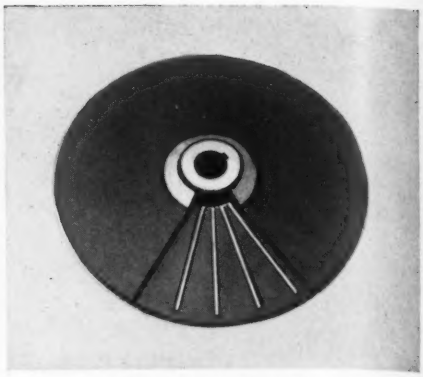

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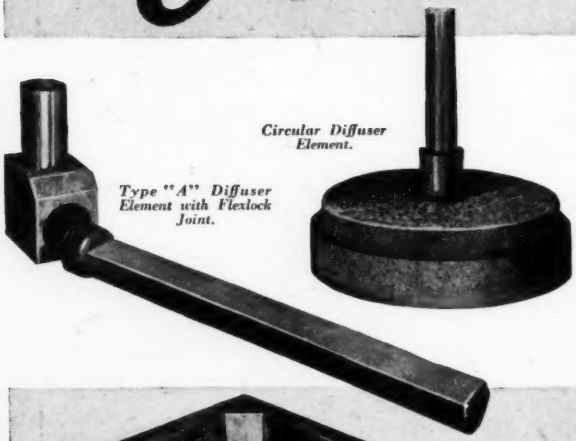

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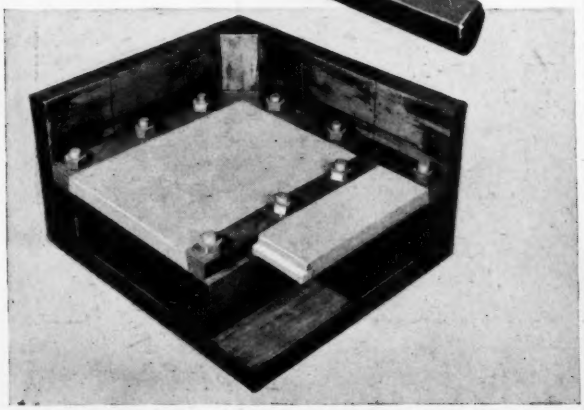

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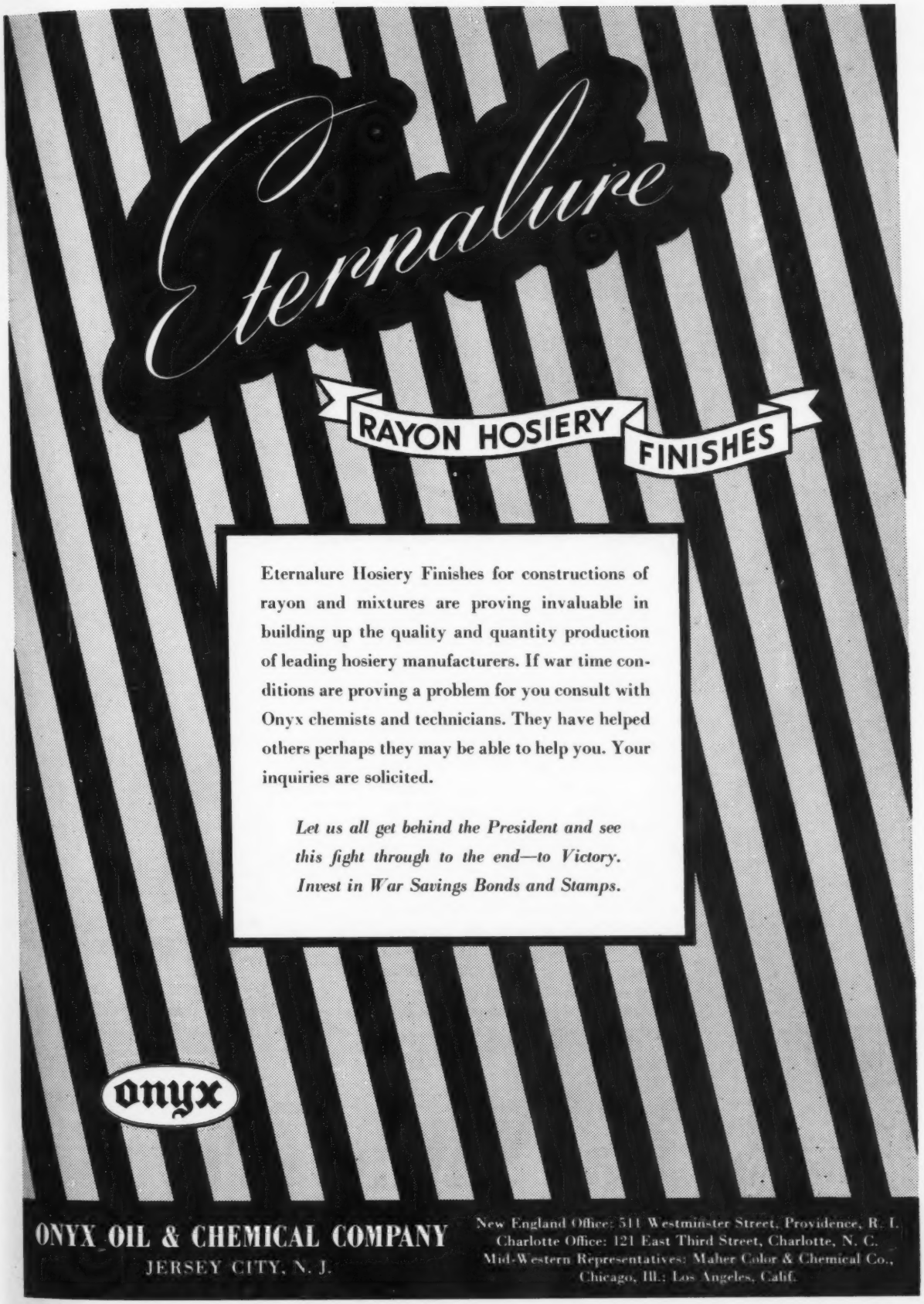
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Contents

Contents of previous issues can be found by consulting the Industrial Arts Index at your library

• Special Articles

Determination of Copper in Textile Materials Treated to Prevent Mildew 395
Carroll L. Hoffpauir and Robert T. O'Connor

Oxidation of Cellulose: The Reaction of Cellulose with Periodic Acid 399
Henry A. Rutherford, Francis W. Minor, Albert R. Martin and Milton Harris

The Analysis of Cation-Active Softening Agents..... 409
Daniel Geltner and W. W. Razim

• Departments

Trade Notes—New Products..... 415

• Proceedings of the American Association of Textile Chemists and Colorists

Open Forum Meeting on Dyeing and Finishing of Army Goods P402

Meeting, Piedmont Section..... P403

Membership Applications P407

One Hundred and Thirty-first Meeting of the Research Committee P408

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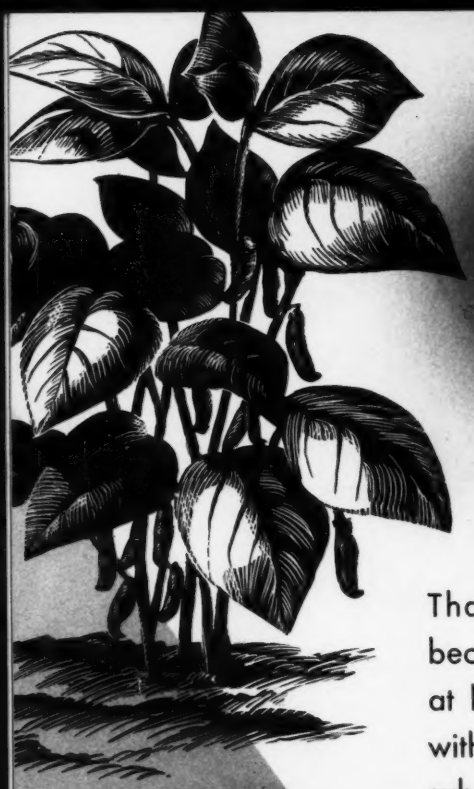

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AMERICAN
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VOLUME 31 AUGUST 17, 1942 NUMBER 17

Determination of Copper in TEXTILE MATERIALS Treated to Prevent Mildew

CARROLL L. HOFFPAUIR and ROBERT T. O'CONNOR

Southern Regional Research Laboratory, Bureau of Agricultural Chemistry and Engineering, U. S. Department of Agriculture, New Orleans, Louisiana

THE need of a method for the determination of copper in fabrics which have been treated with copper naphthenate or other copper containing preparations to prevent mildew was pointed out in a recent issue of the *Textile World*[1]. According to this article, an invitation to bid issued by the War Department specified a sandbag fabric containing between 0.8 per cent and 1.0 per cent copper, but gave no method of determination. A later issue[2] of *Textile World* reported that R. A. Allen had suggested a method, but no supporting data were given. The Analytical, Physical Chemical and Physical Division of this Laboratory has been analyzing a large number of impregnated fabrics for copper by two methods which appear to be well adapted to routine testing of such material. The purpose of the present paper is to give the details of these methods and present data in support of their reliability.

There are methods[3, 4, 5] for the determination of very small amounts of copper in fabrics. Most of these methods are applicable to relatively narrow ranges of concentration. The copper content of the solution actually measured should be between 0.1 and 1.0 part per million in the sodium diethyldithiocarbamate method. The potassium ferrocyanide method measures only a slightly more concentrated range. Adaptation of these methods to the determination of copper in the amounts found in "mildew-proofed" fabrics involves the use of small aliquots followed by relatively large dilutions and further aliquoting. This procedure is wasteful both in time and effort and reduces the accuracy attainable.

Two of the available methods for determination of copper seemed adaptable and have been applied successfully: (A) an iodimetric determination of copper based on the reaction of the cupric ion on potassium iodide to produce iodine, which is determined volumetrically, and (B) a spectrophotometric method based on the measurement of the intense blue color produced by the reaction of the cupric ion and ammonium hydroxide. Using 0.01N sodium thiosulfate solution, the volumetric method will measure from 3 to 60 mg. of copper. This represents a range of 0.3 per cent to 6.0 per cent copper in a 1 gram sample. The spectrophotometric method will measure as little as 0.2 mg. of copper if the volume is held to 10 ml., instead of the 50 ml. indicated in the description of the method; thus a sample as small as 0.05 gm. might be analyzed if a representative sample could be obtained. Details of the methods used are as follows:

METHODS

Reagents

A. Potassium iodide—solid or 50 per cent solution.

B. Standard copper solution—about 0.6 gram of electrolytic copper was weighed accurately and dissolved in nitric acid. Sulfuric acid was added. The solution was boiled until sulfur trioxide fumes were evolved; 50 ml. of water were added and the boiling continued for 10 minutes. After cooling, the solution was diluted to 500 ml.

C. Sodium thiosulfate solution (0.01N.) This solution was standardized against the standard copper solution by the same procedure as was used in the determination.

D. Starch indicator—One gram of soluble starch[6] was suspended in a few ml. of cold water and added to 100 ml. of boiling water. Boiling was continued for 3 minutes.

E. Potassium thiocyanate—5 per cent solution.

F. Ammonium hydroxide—concentrated.

G. Ammonium hydroxide—8M.

H. Potassium fluoride.

I. Acetic acid—glacial.

J. Nitric acid—concentrated.

K. Sulfuric acid—concentrated.

L. Bromcresol-green indicator—0.1 gm. dissolved in 14.3 ml. of 0.01N NaOH and made to 250 ml.

Preparation of Sample

The fabrics to be tested were cut into strips approximately one-fourth inch wide. In order to obtain representative sampling, several strips were taken from different parts of the material to be analyzed. Approximately 1

gram or enough to contain at least 3 mg. of copper was accurately weighed into Kjeldahl flasks (800 ml.). After measuring 5 ml. of concentrated nitric acid into each, the flasks were allowed to stand for 10 minutes in order to partially digest the material. Concentrated sulfuric acid (10 ml.) and three glass beads to prevent bumping were added and the flasks placed in a digestion rack. Low heat was applied at first until the initial tendency to froth had passed (about 5 minutes). The heat was increased and the digestion continued until white fumes of sulfur trioxide were evolved. When charring occurred, due to incomplete digestion, the flasks were allowed to cool somewhat, additional 5 ml. portions of concentrated nitric acid were added, and the digestion repeated until the solutions remained clear. In order to insure complete digestion, the heating was continued for at least a half hour after the solutions became clear. The flasks were allowed to partially cool and approximately 50 ml. of water were poured down the neck and the flasks swirled. The solutions in the flasks were boiled for about 10 minutes and transferred to Erlenmeyer flasks (or volumetric flasks if aliquots were to be taken).

Volumetric Determination

Two drops of Bromcresol-green indicator were placed in each solution or aliquot[7]. The solution was adjusted to about pH 4 by the addition of concentrated ammonium hydroxide until the solution was a faint green. (If preferred, a comparable pH may be obtained by neutralizing with ammonium hydroxide until the odor of ammonia is faintly apparent and then adding 3 ml. of glacial acetic acid.) When iron was present, as indicated by the formation of brown ferric hydroxide on the addition of the ammonium hydroxide, 2 grams of potassium fluoride[8] were introduced and the solution was boiled. After cooling, the iodine released by the addition of 3 to 5 grams* of potassium iodide was titrated with 0.01N sodium thiosulfate solution. When the iodine color had almost disappeared, 1 ml. of starch indicator solution was added. The titration was continued until the starch iodide color disappeared for at least 30 seconds. The end point was sharpened by using about 2 ml. of 5 per cent potassium thiocyanate solution near the end of the titration[9]. Caldwell says that shellac may also be used to sharpen the end point[10].

The iodimetric method for the determination of copper is well known[7, 11, 12, 13, 14, 15]. Scott[11] says: "Only a few elements interfere, such as selenium, trivalent arsenic, antimony, iron, and hexavalent molybdenum. Chromium forms an insoluble sulfate which holds copper. Vanadium interferes." Usually, none of these, with the possible exception of iron and chromium, is present in materials treated to prevent mildew. Iron does not interfere if fluoride is added to form a complex[8] (see Table I). The samples so far encountered have not contained chromium and the method here presented makes no provision for interference by this element. In the presence of chromium, the applicability of this method would need to be established.

TABLE I
Recovery Data

Sample	*Mg. of Copper Present*	*Mg. of Copper Found*	
		Volumetric	*Spectrophotometric*
0.8231 gms. cellulose + 10 ml. copper soln...	12.06	12.03	12.1
0.8360 gms. cellulose + 10 ml. copper soln...	12.06	12.06	12.1
1.0049 gms. of mildew proofed cotton containing 1.71% copper + 5 ml. copper soln.....	23.22	23.02	23.0
1.0029 gms. of mildew-proofed cotton containing 1.71% copper + 5 ml. copper soln.....	23.18	23.10	23.0
1.0028 gms. cellulose....	0	0	0
1.0016 gms. cellulose....	0	0	0
10 ml. copper soln.....	12.06	12.02	12.1
10 ml. copper soln.....	12.06	12.04	12.1
10 ml. copper soln. + 0.01 gm. Fe+++..	12.06	12.06	
10 ml. copper soln. + 0.10 gm. Fe+++..	12.06	12.09	

SPECTROPHOTOMETRIC DETERMINATION

The copper was also determined spectrophotometrically. Aliquots of the solutions of digested material containing 1 to 5 mg. of copper were added to 50 ml. volumetric flasks. The solutions were barely neutralized with concentrated ammonium hydroxide and 25 ml. portions of 8M ammonium hydroxide were then added. The flasks were filled to the mark and mixed thoroughly. The 5-cm. absorption cells of a spectrophotometer were filled and the transmissions at 635 mμ were read against similar cells containing all reagents except copper. The transmissions were converted to extinctions ($\log_{10}$ Io/I, where Io is the intensity through the cell containing the solvent and I the intensity through the cell containing the solution) and concentrations of copper read from the working curve.

The intense blue color developed when ammonium hydroxide is added in excess to a solution containing copper is one of the first color reactions encountered in analytical chemistry and was used over a century ago[16]. Recently J. P. Mehlig[17] made a complete spectophotometric study of the cupric ammonium complex system, including the effects of 60 common ions, the effect of the concentration of ammonium hydroxide, the applicability of Beer's law to the system, and the limits of the color reaction in determining small concentrations of copper. The cupric ammonium system was selected for the spectrophotometric determination of copper in the present study because it seemed to be particularly applicable to the concentrations generally encountered in the analysis of "mildew-proofed" fabrics.

The copper determinations were made with a Cenco-

*It was found that if less potassium iodide was added, a recurrent end point was obtained, making accurate titration impossible.

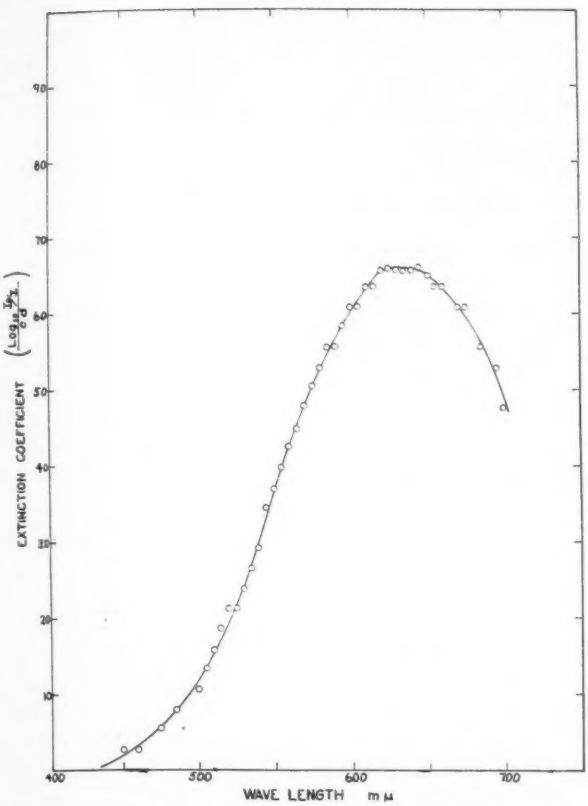

Fig. 1—Absorption Spectra of Copper Ammonium Solution. 4 M NH_4OH

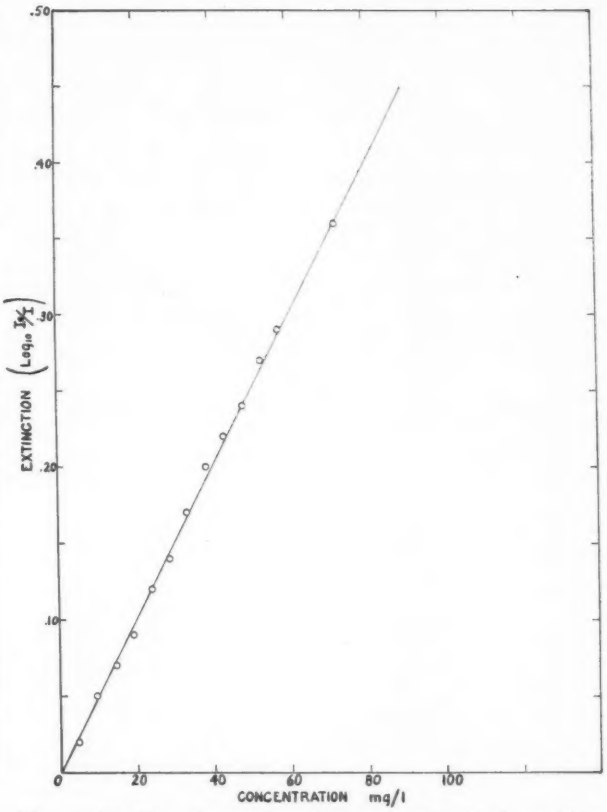

Fig. 2—Working Curve for Copper in Cupric Ammonium System at 635 mμ.

Sheard Spectrophotelometer, using a 5-cm. cell and a spectrally isolated band width of 6 mμ. The present work confirms the conclusions of Mehlig[17] and others[18, 19] that the wave length positions of maximum absorption increase as the concentration of ammonium hydroxide increases. The determinations were made both at the wave length setting 620 mμ (ammonium hydroxide 3M) used by Mehlig and at 635 mμ (ammonium hydroxide 4M). There appears to be no particular choice between the two conditions and presumably the 1.5M ammonium hydroxide recommended by Snell, when readings are made at the appropriate wave length setting of maximum absorption for this value of the pH, would give equally satisfactory results. However, it is important that the concentration selected when preparing the working (or calibration) curve from a series of standard copper solutions be used throughout the analysis of the unknowns. The absorption curve for a solution of copper in approximately 4M ammonium hydroxide (Figure 1) shows the wide absorption band at 635 mμ. The extinction coefficient, i.e., extinction reduced to unit concentration, c, expressed in gms. per liter, and to unit cell length, d, in cm., was plotted against the wave length in mμ to obtain this curve. Figure 2 is a working (or calibration) curve obtained with the spectrophotometer in this laboratory from a series of standard copper solutions in 4M ammonium hydroxide at a wave length of 635 mμ. This curve shows that Beer's law holds for the system up to a concentration of at least 100 mg. per liter in agreement with the findings of other investigators[17, 19, 20, 21]. While the working curve can be extrapolated to pass through the zero of both coordinates, actually it was found, in agreement with Mehlig[17] and Yoe and Crumpler[21], that the method is not applicable to the determination of concentrations of copper less than 10 mg. per liter. The claims of Snell[4] that when 0.01274 mg. of copper per ml. is present, the addition of 0.000016 mg. of copper can be detected, could not be confirmed. Other than to verify the absence of those ions reported by Mehlig to interfere, no studies were made on the effect of extraneous ions on the determination of copper. Many of the samples submitted for spectrophotometric determination of copper were found to yield slightly turbid ammoniacal solutions, due probably to small amounts of insoluble silica. The effect of very slight turbidity on the accuracy of the spectrophotometric measurements has been discussed by Miller[22]. Filtering the solutions through filter paper was found to give more consistent results which were, however, constantly low, due probably to reaction of the cupric ammonium complex with the paper[11]. The procedure adopted was to filter the solution through fritted glass or asbestos, making sure that the clear solution necessary for accurate spectrophotometric measurements was obtained before making the readings.

RECOVERY OF COPPER ADDED TO CELLULOSE AND COMPARISON OF METHODS

In order to check the accuracy of the methods here described, samples were prepared and tested by the procedures outlined; these samples included known copper alone, known copper in the presence of cellulose, cellulose alone, and copper added to samples previously analyzed for copper by both volumetric and spectrophotometric methods. Results are shown in Table I. The recoveries indicate that both methods are accurate within the small experimental error of final measurement.

In Table II are presented percentages of copper in fabrics of both cotton and jute treated for mildew resistance. These values were taken at random from determinations which were made by both methods on aliquots of the same solution from the digestion procedure. They show good agreement between the two methods and indicate that either method is satisfactory. The percentage of copper found in each case was in keeping with that expected, based on the take-up of the fabrics. It is to be noted that even though several different methods were used in producing the mildew resistant fabrics, the copper content was determined satisfactorily.

TABLE II
Comparison of Results Obtained by Volumetric and Spectrophotometric Methods on Duplicate Samples of Cloth

Material	*% Cu in sample determined volumetrically*	*% Cu in sample determined Spectrophotometrically 635 mu*
Cotton osnaburg treated with Cu compounds from ammoniacal solution	0.70	0.65
	0.67	0.67
Burlap treated with Cu compounds from ammoniacal solution	0.71	0.67
	0.70	0.71
Cotton osnaburg treated with copper naphthenate emulsion	0.44	0.44
	0.43	0.43
Cotton osnaburg treated with copper compounds from ammoniacal solution	1.72	1.74
	1.71	1.67
Burlap treated with copper compounds from ammoniacal solution	1.89	1.85
	1.87	1.85
Burlap treated with copper naphthenate from organic solvent; creosote present	0.87	0.80
	0.86	0.90
Cotton osnaburg treated with copper naphthenate from organic solvent	0.46	0.44
	0.46	0.47
Burlap treated with copper naphthenate from organic solvent	1.41	1.39
	1.38	1.36
Cotton osnaburg treated with copper naphthenate from organic solvent	1.49	1.43
	1.49	1.51
Burlap treated with copper naphthenate from organic solvent; creosote present	0.53	0.48
	0.52	0.49

SUMMARY

Two well known methods for the determination of copper have been adapted to the analysis of "mildew-proofed" fabrics. The values obtained by these methods are in good agreement.

The procedure of preparing the copper solution is the same in both methods except for the final step. The actual measurement of the copper in solution consumes only a small portion of the total time required in the determination. The volumetric method has proved satisfactory for routine examination of samples of ordinary size containing moderate amounts of copper (3 mg. to 60 mg. of copper per gram of sample). It is rapid, accurate, requires only ordinary analytical apparatus and is well suited to routine work.

The spectrophotometric method gives an excellent check on the volumetric method and is particularly useful when the size of sample is small or the percentage of copper is relatively low (minimum of 0.2 mg. copper). It has the disadvantage of requiring special apparatus, but it may afford some economy of time in the measurement of copper in solution.

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OXIDATION OF CELLULOSE

The Reaction of Cellulose With Periodic Acid

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ALBERT R. MARTIN, and MILTON HARRIS*

ABSTRACT

The potential scarcity of textile materials in the present emergency makes it more important than ever that adequate means be taken to prolong their usefulness. The useful life of a textile material does not begin until the fibers of which it is made have undergone a series of mechanical and chemical treatments, some of which tend to reduce the strength of the material. Obviously, it is desirable to keep this loss at a minimum. It is well known, for example, that unbleached cotton goods have an undesirable yellowish tint. When such goods are treated with mild oxidizing agents, such as dilute solutions of sodium hypochlorite or hydrogen peroxide, the yellow color is removed. While care is taken to avoid damage to the goods, during this operation, there may occur some loss in strength. Adequate control to prevent this type of damage requires a thorough knowledge of the conditions which produce it. Accordingly, the Research Associates for the Textile Foundation at the National Bureau of Standards have begun an investigation of the way cellulose is attacked by oxidizing agents.

The first reaction studied was that of cotton cellulose with periodic acid. Periodic acid is not used in bleacheries for processing cotton, but the known selective action of this reagent made it desirable as a first subject of study in order to form a background for the proper understanding of the action of the common bleaching agents. It was found that in some cases, the action of this oxidizing agent does not result in a direct loss in strength but rather a latent type of damage is produced. Such damage is not indicated by usual strength tests until the bleached product is laundered or otherwise subjected to alkaline solutions. It appears that, in such cases, the chemical stability of the cellulose molecules of the cotton has been reduced to an extent which does not appreciably impair the original strength of the material. Further treatment of the chemically unstable groups with alkalies results in far-reaching alterations in the molecular structure, thereby producing the loss in strength.

I. INTRODUCTION

CELLULOSE can undergo many modifications in oxidizing media and accordingly the properties of oxidized celluloses, generally referred to as oxycellulose, vary widely. It appears that the attack on cellulose by oxidizing agents is principally confined to three points namely, (1) the aldehyde end-groups, of which there are few, if any, in native cellulose[1] and which can be oxidized to carboxyl groups; (2) the primary alcohol groups which can be oxidized to the aldehyde or carboxyl stage; (3) the glycol group (the 2, 3-dihydroxy group) which can be oxidized to the ketone, aldehyde or carboxyl stage. Until recently, it has generally been assumed that the oxidative attack occurred principally at the primary alcohol groups, a hypothesis which gave rise to considerable confusion. This hypothesis did not adequately explain the different behaviors of oxycelluloses prepared with various oxidizing agents. Thus, whereas Clibbens and Ridge[2] were able to show that treatment of cotton with any acid resulted in the same corresponding increase in cuprammonium fluidity and decrease in tensile strength, a similar general relationship did not exist for all types of oxycellulose. The data in Table 1 (from reference 2) illustrate this point. This apparent anomaly was indeed disturbing, since both the fluidity and strength of cellulosic materials are regarded as a function of molecular chain length.

TABLE 1

Relation between cuprammonium fluidity and tensile strength of various hydrocelluloses and oxycelluloses

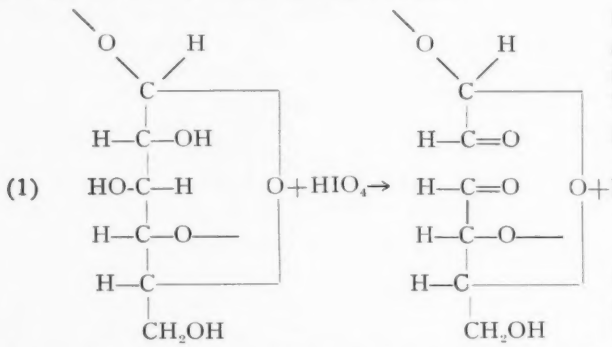
Cuprammonium fluidity rhes	*Loss in tensile strength resulting from: Treatment with any acid percent*	*Treatment with $K_2Cr_2O_7$ + $(COOH)_2$ percent*	*Treatment with NaO Cl percent*
10	10	1	7
20	34	6	25
30	58	16	47

As a solution to the difficulty, Davidson[3, 4] made the important suggestion, which he later supported by extensive experimental work[5], that some types of oxidation do not result in direct scission of the chain molecules but reduce the chemical stability of some of the linkages in in the cellulose chain towards alkali. Types of oxidation which do not rupture the chains would have little or no effect on strength, but if these types of modification result in a material which is unstable towards alkali, the fluidity in the alkaline cuprammonium solution will be high. This then would explain the apparent lack of an identical

*Research Associates of the Textile Foundation at the National Bureau of Standards.

correlation between strength and fluidity for all types of oxycelluloses similar to the correlation existing for all types of hydrocelluloses.

Davidson's hypothesis was lent considerable support by the investigations of Jackson and Hudson[6], who showed that in the oxidation of cellulose by periodic acid, cleavage of the glucose ring between carbon atoms 2 and 3 occurred with the conversion of the secondary alcohol groups to aldehydes, according to the following scheme:



A reaction of this type does not result in a cission of the molecular chains, and hence little, if any, loss in strength occurs.

While it appears that periodic acid (at least during the early stages of oxidation) and a few other oxidizing agents may be quite specific in their attack on cellulose, the action of many oxidizing agents to which cellulose is frequently exposed is more complex and less well understood. A major difficulty in attempts to ascertain the exact course of the various types of oxidation has been the lack of analytical methods of sufficient specificity and sensitivity to determine quantitatively the various groups which may be formed and to allocate them to their proper positions in the glucose residues. Recently, however, a number of new methods have been developed and accordingly a series of studies of the course of oxidation of cellulose by various reagents was undertaken. The present paper reports the results obtained with periodic acid.

II. EXPERIMENTAL PROCEDURE

1. Preparation of Materials

Raw cotton was dewaxed by extraction with hot alcohol for 24 hours and then washed thoroughly with cold distilled water. The cotton was further purified by boiling with a 1 per cent solution of sodium hydroxide for 8 hours; the procedure was essentially the same as that described by Corey and Gray[7] except that the apparatus of Worner and Mease was used[8].

The oxycelluloses were prepared by steeping samples of the purified cotton in solutions of periodic acid buffered with sodium acetate at pH 4.6. The extent of oxidation was controlled by varying either the concentration of periodic acid or the time of treatment. In most cases, however, a concentration of about 0.025 *N* periodic acid was employed. All treatments were carried out with the ratio of cotton to solution of 1 to 50 and a temperature of 25° C. ± 0.1°. Immediately following the oxidation, the samples were rapidly washed in cold distilled water until a test of the washing for periodate was negative. The materials were then conditioned in the dark, in a room maintained at a temperature of 21° C. and 65 per cent relative humidity. Under these conditions, it was found that the properties of the oxycellulose did not change over prolonged periods of time.

2. Methods

The concentration of periodic acid was determined by titration of an aliquot of the solution with sodium arsenite after the addition of sodium bicarbonate and potassium iodide, according to the method of Muller and Friedberger[9]. The number of milliequivalents of oxidant consumed per gram of dry cellulose was calculated from the difference in the amount of arsenite required by a control solution, and by an identical solution containing the sample.

The aldehyde content of the oxycellulose was determined by a modification (described below) of the iodometric method as used for hydrocellulose by Martin, Smith, Whistler, and Harris[1]. This determination depends on the oxidation of the aldehyde groups to carboxyl groups by iodine. The amount of iodine consumed by the oxycellulose was determined as follows: 10 ml. of a 0.05 *M* sodium borate solution having a pH of 9.2 at 25° C. was pipetted into a 125-ml. glass-stoppered Erlenmeyer flask and then placed in a thermostat at 25° C. 20 ml. of a 0.03 *N* iodine iodine solution containing 20 gms. of potassium iodide per liter, previously brought to 25° C. was then added and approximately 100 mg. of the sample to be titrated immediately introduced. (With this amount of sample, it is important that the oxycellulose contain no more than 0.15 millimoles of aldehyde groups per gram; the ratio of milliequivalents of iodine to milliequivalents of aldehyde should not be below 25). After 6 hours, 15 ml. of 0.1 *N* HCl was added and the iodine remaining in the solution was measured by titration with standard sodium thiosulfate (about 0.013 *N*) with starch as the indicator. Blank determinations on identical solutions which did not contain cotton were made. From the difference in amount of thiosulfate required by the blank solution and by the solution containing the sample, the milliequivalents of iodine consumed per gram of dry sample were calculated. As explained elsewhere[1] a correction for the iodine consumed by purified cotton in the same time interval must be applied in calculating the aldehyde content of the oxycellulose.

An independent check on the iodine method was made by analyzing for carboxyl content after oxidation of the aldehyde groups with chlorous acid. This substance is particularly useful for this purpose in view of the selective action towards aldehyde groups observed by Jeanes and Isbell[18] in their work on the oxidation of certain aldoses. A 0.4 *M* solution of chlorous acid was prepared immediately before use by the acidification of a solution of technical sodium chlorite to pH 2.5 with the appropriate quan-

(Continued on page 412)

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Open Forum Meeting on Dyeing and Finishing of ARMY GOODS

Conducted by Piedmont Section, June 20, 1942

AN open forum meeting on the dyeing and finishing of army goods was conducted on June 20th by the Piedmont Section in Charlotte, N. C. The discussion was led by Dr. R. E. Rupp of Pacific Mills. The report of the meeting follows:

Dr. Rupp: The subject of tonight's meeting is "Army Fabrics: How We Can Do Them Better." I have a number of questions which have been handed to me; I will read them. The first one is: "In dyeing herringbone twill, what preparation should the goods have before dyeing?"

. . . There was no discussion on this subject and Mr. Rupp continued . . .

WATERPROOFING RAINCOATS

Dr. Rupp: I have a question of my own that I would like to ask: There are a lot of raincoats being dyed and, beginning recently, plastics have been used instead of rubber for the coating. The waterproofing that is ordinarily put on the raincoat materials is entirely satisfactory for rubber coating but, turning to the plastics, the waterproofing compound is not satisfactory. What sort of waterproofing is to be used?

. . . Major Frank Steadman of the Philadelphia Quartermaster Depot was called on to answer this question . . .

Major Steadman: The matter of water repelling or better, water repellant finishing of raincoat fabrics has been of questionable efficiency. However, a certain percentage, at least, of that finish was carried through the rubberizing process, either the calender coating or the spread coating, in which latter case the rubber compound is applied with a solvent.

Now that we have gotten out of the rubber and gone over into synthetics, vinyl polymers and co-polymers, those being polyvinyl chloride and co-polymers of vinyl chloride and vinyl acetate or the polyvinyl butyrals for the spread coating, much stronger solvents are used. Cyclohexanone and methyl ethyl ketone are two of the principal solvents.

I have heard no complaints that the proofing does not carry through. It may be that we have not been examining that too critically; that may be the reason that we have not had the complaints in the research section. But those are the two principal solvents that we are going to meet up with.

When you go into polyvinyl butyral, some of the more common solvents can be used. I believe acetone is one of those that can be used. In any event you are up against a different set of solvents, and, in general, stronger and more active solvents than you meet in the rubberizing game.

I believe that with those firms who are proofing the goods by calendering either polyvinyl chloride or vinyl butyral that you will probably have no difficulty, unless you come up against the proposition of the coaters securing adequate adhesion. I know of at least one firm that is having some difficulty in getting adequate adhesion, in attempting to apply a comparatively light coating of polyvinyl chloride on a balloon cloth which we supplied in the gray for this firm to have processed and printed, preparatory to the coating of the fabric. Now, they have attempted to use still another coating and that is the synthetic rubber of the butadiene type. I believe they are applying that as a spreader coating.

But this opens up an entirely new field, which will probably have to be worked out quite largely between any of you who are processing those goods for the coaters and yourselves as finishers. But I will say this: that, so far as I can see, the use of the synthetic rubbers and resins for certain lines of the services will continue. For some purposes the cloth will probably be used in a semi-prepared state, and there will be continued use of dyed fabrics coated principally with the copolymer commonly designated Vinylite VYNS and the polyvinyl butyrals.

Those materials are going to continue to be your proofing materials for quite some time; so you may as well prepare yourselves—if you are not already prepared—to handle materials for those coatings.

Dr. Rupp: Aluminum acetate was very satisfactory for rubber but, because of the solvents which are in these synthetic resins it is evidently not satisfactory. I think the answer to it is: Waterproofings that are not dissolved or acted upon by the solvents of the plastics.

I'd like to ask the Major's opinion of casein. Of the non-soluble materials, casein is one of the best known waterproofing agents. Do you suppose casein would stand up against the waterproofing solvents of the synthetic resins?

Major Steadman: I can't tell you whether casein will stand up or not; I see no reason why it should not—especially if it has been set—it being a resin, itself. It may be that a formaldehyde treatment in combination with the casein resins, may set that; but I have not considered the casein itself as primarily or even secondarily a water-repellent finish.

Photos by Robert W. Philip

Snapped at the Open Forum Meeting, Piedmont Section, June 20, 1942

MEETING, PIEDMONT SECTION

APPROXIMATELY 225 members of the Piedmont Section attended the summer meeting which was held at the Hotel Charlotte, Charlotte, N. C., June 20.

The banquet session was featured by a symposium on the subject of dyeing and finishing problems in relation to the defense effort. Dr. R. E. Rupp, Superintendent of Pacific Mills, took charge of this part of the program. Representatives of the Philadelphia Quartermaster Depot who were present participated in this program. (The stenographic report of this symposium appears on these pages. Ed.)

Respectfully submitted,

D. STEWART QUERN, *Secretary*.

This may or may not force a consideration of new methods of holding water-repellent finishes, or force finishing after the application of resins to the fabric, or it may force the application of the resin to both sides of the fabric and eliminate thereby the applications of any water-repellent finish. That is something which we are rather anxious to avoid if we can—because any coating on the fabric, of course, increases the stiffness.

If we can maintain a single coating of the fabric—a fabric coated on one side only—not necessarily a single process of coating, we will be able to have a softer fabric for the purpose than if we must necessarily go to the double-coated material. But what types of water repellent materials we may be able to use on this, frankly, I don't know.

Mr. Crist: I'd like to ask the Major if his interest in synthetic resins is born primarily from the rubber shortage or from a feeling that we can get superior finishes with the synthetic resins.

Major Steadman: There is, definitely, a shortage of rubber in the country. Crude rubber is being used in those things for which crude rubber only can, at the present time, be used or for which crude rubber is available in sufficient quantity.

BLENDING COTTON AND WOOL

Dr. Rupp: Here is another question: "In blending dyed cotton and dyed wool, for the manufacture of army shirts, in what form should the cotton be delivered to the woolen mills, i.e., raw stock, card sliver, combed sliver, roving, etc.?"

. . . Mr. Cleveland of the Beacon Manufacturing Company was called upon to answer this question . . .

Mr. Cleveland: We have none of that kind of work at our plant. I don't know; it depends entirely upon the fabric that is being manufactured by blending cotton with wool. Our work is all on army blankets and so we don't have any problem of that sort.

Dr. Rupp: Can you tell us about what is being done in the south on woolen fabrics?

Mr. Cleveland: The only thing that I can tell you is what we are doing with Army blankets. There is quite a little work being done in the South; in fact, we have a good bulk of the Army blankets and overcoating—twelve ounce fabrics are being made here in the South—but the great bulk of that work is being done in the East, where there are so many woolen mills. I think most of the work that is being done in the South is largely blankets. I don't know how much of the Navy work is being done here, but

there are a lot of Navy blankets being made and I know quite a few plants are making overcoating. But as to the worsted end of it—shirting and such as that—that is being done in the East, I believe.

WASH FASTNESS OF PIGMENT COLORS

Dr. Rupp: I have another question: "Can the wash fastness of pigment colors, after exposure to light, be improved, or has it been done?"

. . . Mr. Robbins of the Aridye Corporation was called on to answer this question . . .

Mr. Robbins: As you know, pigment dyeing was accepted as an alternate for herringbone twill. We stepped into production, with one plant, and lo and behold, we found out that after it was subjected to violent weather, the ultra-violet rays broke it down. We placed the whole picture before Major Steadman, Colonel Painter, "et al," and, as a result, they agreed with us that it was pretty bad, and for us to do better and do it quick! As a consequence, we made some changes in formulation, which showed improvement; I won't say it was perfect, but it did definitely show improvement.

From there on, we went on with production of a new pigment which we now have, and have been running with for a time, and we feel that it is pretty good. Now, whether it is any good or not—with a bow to Major Steadman, I will have to put it back in his lap; but I will go so far as to say that, with its improvement, it can be made faster to ultra-violet rays than it ever was before. And there we are, today. How good we are, we do not know.

Now, I would be glad to answer any questions on this, if they are not technical. I do have some evidence here which I can't very well pass around, at this time, because there are too many here; but if any of you are interested in it, I would be glad to show it to you.

Dr. Rupp: Mr. Robbins, you are very reassuring.

Mr. Hayes: You referred to a new pigment. Do you feel that it was a molecular breakdown of the pigment?

Mr. Robbins: As I understand, it was. It had the effect of breaking down the pigment, in the absorption of the ultra-violet rays—that's the way I understand it; I don't know, technically.

Major Steadman: As stated, the initial samples and first deliveries, on an actual "wear and use test," did show a definite breakdown. Now, this is something that will land right back in your own laps, though: and that is, that some of the vat-dyed samples were not a whole lot better! (Laughter.)

So it comes right straight into the question of the penetration which is being secured in the vat dyeing of herringbone twill. Maybe we have been too lax, in our acceptance of the herringbone twill—but we needed the goods, and it serves the purpose. But we have had kicks from the field, that the herringbone twill fades too badly; it fades out. Now, we know that good vat dyestuffs are being used on it. Examination of it shows that insufficient penetration has been the cause, all too often; so, not that I am rising particularly to the defense of any corporation which makes a pigmented resin application, but that presents the occasion to bring to your minds the fact that penetration of the vat dyestuffs, on the herringbone, in particular, has been poor. We would like awfully well to see more complete and better penetration. I think probably this group represents a fair percentage of the finishers of the herringbone twill.

Dr. Rupp: I think that pigment dyeing is really on the way! Now, I don't know who is going to put it over but the vat dyestuffs may be as obsolete as the "trolley-car" ten years from now. Now, I have another question...

USE OF SULFUR DYESTUFFS

"Is there any reason why sulfur dyestuffs should not be used?" Now, I promised the Major that I wouldn't bring that up tonight—it was thrown for such a loss this morning. I will pass it over, unless some one says something right quick.

Major Steadman: As I said before, I think there is very little question about the suitability of sulfur dyestuffs, when they are properly applied. Our only question is, how are we going to tell when they are right; or, perhaps, to put it another way, until we can be in the position of your coming to sell us goods, based upon past performance, and not primarily upon meeting a written set of specifications we cannot accept the risk. So it is up to you to finish those properly; or perhaps I should exclude this group and lead the "black sheep" into some other "pasture"; but, until such time as we can know by appropriate test that the sulfur dyed goods which may be offered are not going to tender, through improper dyestuffs or improper finishing, we cannot recommend an action which is going to place the "contracting officer" in a position where he himself will be personally financially responsible in the event that those goods at a future date tender and become worthless.

Now, I mentioned that briefly, at the Greenville meeting, in January; I don't know whether I made the point clear or not; but there is just that personal financial responsibility of a contracting officer, in the Army. They maintain records for a long time, and can trace things back a long way, and they don't hesitate in the least to clamp down on any one who may be culpable, who may have fallen down on his job. For instance, I know of one or two Field Artillery officers who, somehow or other, failed to make a thorough check on every possible contingency that might arise in connection with some firing—and the Lord knows there are enough of them!—but who had a gun blow up. They *paid* for the guns! Now, that's how hard-boiled the Army can be!

So with a personal liability like ours, we are not going to recommend a procedure until we know we are on safe ground, for all procurements—not merely for procurements

from reputable firms, but for *all* procurements, from "Tom, Dick and Harry" and "Tom Jones down the river"!

That is the situation. I am anxious to see an answer to this question which is being studied, of a suitable and adequate simple test to determine whether or not the sulfur dyestuffs have been properly applied, using proper materials in such a manner that there may not be a back-fire at a future date, if improper materials have been used or if poor storage conditions arise.

Now, that is our side of the picture, on sulfur dyestuffs and their use. Otherwise, I see no reason why they should not be used, except for that "bug-a-boo," which is well covered in official communications relating to the deterioration of certain materials which had been accepted —and I think most of you know that our Inspection Section up there is not too lenient. This particular batch of goods went through, anyway, and they turned up, under fairly decent storage conditions, badly tendered. Well, now, Washington has not forgotten that. And so we are going to have some "ammunition" back of us, before any of the Technical Personnel will recommend any use of materials on which so black a history exists.

Now, I know that a lot of you are under difficulties, in the application of the direct dyes in meeting requirements of color fastness on linings, comforter covers and on sox. I wish we could get out of those difficulties, on those materials! That is our position.

SIZING HERRINGBONE TWILLS

Dr. Rupp: Major, I was *afraid* you were going to work around to that: Well, we don't all finish goods. I have heard that there are some cotton goods woven in the South—and I have a question in front of me which demands an answer from an experienced cotton mill man: "What size compound should be used in the weaving of herringbone twills which will make a finisher's job a 'bed of roses'?"

... Mr. Murphy was called on to answer this question ...

Mr. Murphy: Mr Chairman, we do not finish them; we pass them to the Proximity Mills, and these gentlemen finish them. We use a "size" that can be taken out of the warp, before they are finished; and anything that contains a gum is very detrimental. We try to stay away from that, and try to put just as little starch in the warp as we possibly can. We usually "get by" with about seven per cent of "size"; we have some shading, but we find that it helps the finishing a great deal, not to put gums in our "sizing."

Dr. Rupp: Size them, but don't put any more starch in them than you have to.

Mr. Murphy: Just a little.

WEATHERING

Dr. Rupp: I'd like to ask what "fastness of ten days' weather" really means?

Major Steadman: "Ten days' weather" means the ten-day weather test exposed approximately forty-five degrees from the horizontal, facing the South, in December or in June—whenever the material may arrive to be tested. That is a test I have been concerned about. I am afraid—rather inaccurately—but it means ten days' exposure to the weather,—whatever may come—in wind, rain, fog, snow, sleet, or in a blazing hot sun like we had this afternoon—whatever comes.

I have discussed briefly, with some members of the Association, the possibility of a sliding scale, based upon the latitude at which the test is to be conducted, and the period of the year, as to the time for a basic weather exposure.

Now, that is mentioned, just as food for thought. If there are any of you here who might be interested in that particular subject and may want to conduct any investigations about that, I would be very happy to collaborate with anyone on it. Maybe I shouldn't mention the possibility of there being varying climatic conditions or varying atmospheric conditions as between industrial sections, with a weatherboard set up in the lee of your power plant, or one set up, say, twenty miles out in the country, on a farm, away from any oil-burning or coal-burning equipment. But, as I said before, that is only a suggestion; those are just a few thoughts on that test that are passed out for your consideration.

Mr. Thompson: Does the Quartermaster Corps recognize, or are they giving any recommendation, to any degree, to such a test as we perform in the Weatherometer, as a satisfactory test for the weather fastness?

Major Steadman: Neither the Weatherometer nor the accelerated fading units have been accepted as standard test methods. Some of your Research Committees are, I am sure, fully cognizant of some of the deficiencies in those units. Until such time as those are worked out so that you do get consistent results—quite largely from machine to machine, from unit to unit—not necessarily just within any one unit, but from unit to unit—I don't think that the Quartermaster Corps will be establishing that as a primary fastness test.

Now, I know your Research Committees are working on that question; I don't know how many of you are cognizant of the scope of that question, but it goes so far as involving the question of the effect of the ultra-violet light on the glass globe protecting the samples from the carbon arc. It carries through to that extent; it is known, that after a period of exposure, the transmission characteristics of a glass globe are changed. That may be the extreme of the difficulties involved in that particular test, but it is not the only one.

SAND-BAGS

A Member: What is the purpose of the copper naphthenate on sand-bags—how long is it tested, and what duration must sand-bags stand up under?

Major Steadman: Unfortunately, that question will have to be taken up with the Corps of Engineers who are charged with the procurement of the sand-bags. I don't

know exactly what their specification requirements are, but I understand that they are very strict. From personal observation, I know that sand-bags revetments have been built at various critical points, to protect gun crews—for anti-aircraft—for critical defense of military areas. Those have been up for around six months now, and unless whatever compound is used to make them resistant stands up, we are going to have to use considerable yardage of either burlap, which is a little bit hard to get from India just now, or of osnaburg; and if they do that, it will probably take more of your looms off of more profitable goods. It is just simply to enable those revetments and whatever use is made of the stand-bags, to stand up for a longer period, to conserve the material.

Of course, you probably have the idea—some of you, from previous experience—that labor does not count in the Army; but "man-hours" do count, after all; so it is a conservation of material and man-hours—that is the reason for the mildew-proofing on the sand-bag.

Now, I don't know whether I have answered the question; I know I have not, as regards the specification requirements; but I know it is a question of very distinct interest to the Engineers—and possibly of increasing interest to other branches of the Supply Service.

FASTNESS OF UNIFORMS

Mr. Cleveland: Mr. Chairman, I'd like to ask the Major a question. This is slightly irrevelant; but, in the last war—speaking of the uniform items, the first thing we did, when we were issued any of our outfit, leggings and cotton goods, and so forth, was to go down to the kitchen and get some salt and water, and wash those things out, and let them bleach out as long as they could; the whiter they bleached and the quicker the color came out of them—the more enhanced was our appearance as an "old hand in the Service." And I'd like to ask the Major if Army customs have changed any. (Laughter.)

Major Steadman: I am afraid that question by Mr. Cleveland was directed, not particularly at me, but to some of the dyestuff people. (Laughter.)

That is one reason why we like the vat dyestuffs for the outer items of the uniform, because they will, in general, stand up more uniformly; the boys can't find any tricks by which they can get them out, to look like "old soldiers"! (Laughter.)

DYEING PLY YARN

Dr. Rupp: I have a question: "In dyeing four-ply yarn, should the yarn be dyed in the single, or is it satisfactory to dye the gray yarn in the ply?"

. . . Mr. T. R. Smith of the Wiscassett Mills was called upon to answer this question . . .

Mr. Smith: The only thing that I can tell you is that we are dyeing in single, and plying it afterwards. We have never been able to dye it satisfactorily after it was plied. Now, I would like to see some of you stand up and tell us how it is done; we have never been able to do it, in our plant. I would like to say, for the benefit of you people in Charlotte, there is a plant making webbing, right here in Charlotte, about five blocks from here. F. W. Warrington started the plant, called Marjane Weaving, Incorporated, on Graham Street. I went down there this afternoon and saw it. They made the first cartridge-belt yesterday, dyed single, and plied afterward. I think the four-ply yarn could be dyed in the skein; and another way would be to dye in the raw stock, which is also being done in a large way.

SEWING THREAD

Now, I'd like to ask the Major a question, here, which I think is pertinent; I think there are others who might be interested in this, as well as myself: In dyeing sewing-thread, why does the Quartermaster Corps insist on fast-to-chlorine thread, for woolen blankets? Can you answer that question, Major?

Major Steadman: I'm afraid I can't answer that one, unless it is simply a carryover from the time when we were able to get good vat dyes, well dyed, without any difficulty, without meeting up with priority requirements on chemicals—it is simply a case of taking a good product, rather than one of lesser quality.

On the other side of the picture, I have heard some comments that there is little or no use to permit lesser fastness on sewing-thread—because they might as well dye all the sewing-thread with vat dyes, and, in that way, we get the best we can, all the way through, to avoid having small lots, dyed with the direct dyes or the less fast dyestuffs.

As I say, it is simply a case of taking good fastness wherever we can get them—giving ground only when we feel that we must give, to that extent.

The whole thing is pretty well summed up in these words: It's a case of not going to a lesser quality of materials, where we can avoid it.

Granted, that probably, for the launderings which the blankets will require, there will be very little loss of color in a good direct-colored thread.

On the other hand, some of you have been struggling pretty hard, to get an acceptably fast color with direct dyes, on several fabrics. I don't know whether it would be so comfortable, to go to the direct dyes, compared with the vats, or not.

SULFUR DYED HOSIERY

Dr. Rupp: I have another question here: "What is the objection to dyeing hosiery with sulfur colors?"

Major Steadman: Again, I am going to fall back on the old story—I rather think I answered that question before—when we can test for proper dyeing. If we knew we could get it, it would be all right.

We do know this: that difficulties are being encountered with direct dyes. I was rather expecting a barrage of questions, on how to meet the wash-fastness test. I don't know how it happens that that has not materialized.

But, on the other hand, I say what I think Colonel Painter told some of you, and that is, that some firms are doing an acceptable job with direct colors, on the hosiery.

If we can get to the bottom of the question, on sulfurs, we will take those—it will let you out of an awfully "hot seat," on hosiery, until such time as we can get back into vats on which we know there is no question.

Mr. Smith: About this sulfur dyeing question, most of you who have dyed hosiery, have Monel metal machines, and you would not have machines long, if you dyed sulfur colors in these Monel metal tubs.

Now, I think that the yarn would be all right, dyed with sulfur colors, and the hosiery; but I don't think the machinery would stand the sulfide very long.

FLAME PROOFING

Major Steadman: A problem has been raised very recently—one on which some of you have offered some suggestions, and which we can probably take out of the file. Some of the rest of you may have other experiences and thoughts and suggestions on the same questions; and that is, how to treat a fabric, to make it, if not flameproof, at least highly flame-*resistant*. As you can probably guess, the occasion for that, you probably all remember the so-called Molotov cocktail that we heard about in the Spanish campaign—I think either that item or a modernized version of it is still being used.

So we are interested in flame-retardant finishes for cotton goods, and we would appreciate any suggestions on that—any remarks or experiences which you have had with it—in the form of a letter to the Quartermaster Depot in Philadelphia. It is a call for help, in other words; and if there is anybody who knows anything about it, I would like to have your thoughts on it.

Mr. Niessen: Major, should it be wash-resistant or can it be replaced after each laundering?

Major Steadman: The process should be used on clothing, and probably, desirably, one which could be applied to the clothing after it is made up. It should be wash-resistant or, if not, it should be sufficiently inexpensive and easy to replace in the laundry wheel each time the clothing is laundered or dry-cleaned.

Dr. Rupp: I might suggest one or two that have just come to mind: Chlorinated rubber and paraffin which is wash-resistant and flame-retardant and the stannates. Would either of them answer your question?

Major Steadman: The stannates are interesting, yes. The chlorinated compound is probably ruled out, because of requirements with which those of you who are finishing ducks are well conversant. We will probably have to forego a very serious consideration of the chlorinated rubber.

Another factor militating against their consideration is the fact that they do increase the weight and stiffness which are undesirable since the finish is desired for use on clothing. And so the less change in the characteristics of the fabric, the better it will fill the purpose.

A Member: Is that on woolens or cottons?

Major Steadman: Cottons, primarily.

MEMBERSHIP APPLICATIONS

Senior

Charles Raymond Atkins—Dyer and Finisher, H. R. H. Hosiery Mills, Inc., Moberly, Mo. Sponsors: E. L. Lueck, A. Roy.

Edwin C. Dreby 3rd—Chemist, Scholler Brothers, Inc., Philadelphia, Pa. Sponsors: F. C. Scholler, K. H. Barnard.

Harold C. Knight—Textile Engineer, War Dept., Ordnance Div., Boston, Mass. Sponsors: E. R. Schwarz, C. A. Seibert.

Robert M. Wilson, Jr.—Chemist, National Aniline Div., Allied Chem. & Dye Corp., Philadelphia, Pa. Sponsors: W. Ebersold, F. J. Allen.

Robert S. Withers—Plant Chemist, Gotham Hosiery Co., Philadelphia, Pa. Sponsors: L. S. Zisman, E. A. Demuth.

Frank M. Steadman—Major, U. S. Army, Quartermaster Corp. Sponsors: T. R. Smith, H. C. Chapin.

Associate

John Nerl—Salesman and Demonstrator, American Aniline Products Inc., Cincinnati, Ohio. Sponsors: E. F. Smith, L. B. MacFarland.

Student

Saul Nulman—Bradford Durfee Textile School. Sponsor: L. B. Coombs.

Corporate

Greenville Finishing Co., Greenville, R. I.

Senior

John F. Hardy—Dyer, Medlicott Co., Windsor Locks, Conn. Sponsors: F. W. Swan, F. M. Bonnett.

Ralph A. Nelson—Chief Chemist, Velveray Corp. Sponsors: A. N. Graves, J. H. Kilcommons.

Paul H. Netherwood—Chemical Engineer, Sprague Specialties Co., North Adams, Mass. Sponsors: D. G. Spink, D. deGoenoz.

Frank Albert Stetkiewicz—Head Dyer, Newport Finishing Corp., Quidnick, R. I. Sponsor: J. H. Gormley, Jr.

Junior

Joseph Smith—Laboratory Asst., Pacific Mills, Worsted Div., Lawrence, Mass. Sponsor: F. J. O'Neil.

Associate

Michael P. Mulqueen—Research Director, Hatters' Fur Exchange, Inc., Walden, N. Y.

Student

John C. Rockett—Bradford Durfee Textile School. Sponsor: L. B. Coombs.

Application transfer to Senior Membership. R. G. Yeaman.

ONE HUNDRED AND THIRTY-FIRST MEETING OF THE RESEARCH COMMITTEE

THE 131st meeting of the Research Committee followed the Council meeting in New York on Saturday, June 27, 1942. Present were Chairman Louis A. Olney presiding, A. I. Anderson, K. H. Barnard, W. H. Cady, H. Christison, R. F. Culver, J. N. Dalton, R. E. Derby, C. Z. Draves, W. Ebersold, O. G. Edwards, A. H. Gaede, S. L. Hayes, N. A. Johnson, A. E. Jones, D. R. Kneeland, D. P. Knowland, H. G. Macdonald, O. F. Marks, J. F. McCoy, J. E. Meili, J. A. Mellor, W. R. Moorhouse, G. A. Moran, C. L. Nutting, F. A. Prisley, R. Robertson, B. A. Ryberg, C. A. Seibert, J. Shanks, T. R. Smith, R. H. Souther, F. M. Steadman, B. Verity, W. v. Bergen, P. J. Wood and H. C. Chapin, Secretary.

There was further discussion of the work on olive drab wool fabrics, by Major Steadman and other members of the sub-committee. Mr. Ryberg reported progress on correlation of standards by the American Standards Association Committee on Fastness of Colored Textiles. Thomas R. Smith reported for the Committee on Sulfur Dyes, outlining efforts at agreement upon an accelerated test for effect of age on fabric strength.

Reporting on fastness to washing of rayon, Mr. Knowland described the accumulation of a variety of commercial samples for determination of representative behavior. Mr. Moran showed results of many Launderometer tests on printed fabrics, illustrating the different behavior of dyes and pigments, and the need of very careful adjustment of rubbing action in the test to make it comparable with actual laundering. The effect of light exposure in conjunction with laundering was discussed. Arthur I. Anderson, newly appointed representative of the American Institute of Laundering on the Research Committee, discussed the difference in point of view of the laundryman as contrasted with that of manufacturer and dyer, citing among other things, the necessity of giving a light shade more punishment in laundering in order to make it appear clean.

Mr. Ryberg described modification of the perspiration test, involving gradation of time, and matching degree of discoloration against standard grays. Cards of twelve Munsell grays for this and other purposes were shown, available from the Association for three dollars each.

Organization of a Committee on Tests for Mildew Proofing was announced, with William G. Chace as Chairman. A report was read from A. R. Macormac, Chairman of the Committee on Testing Fire Resistant Material, and the importance of textile fireproofing in modern warfare was emphasized by Major Steadman. Mr. Marks reported for the Committee on Shrinkage of Rayon Fabrics; and Mr. Seibert for those on Fastness to Dry Cleaning, and Fastness to Chlorine, with statement that the Quartermaster Corps had approved the chlorine test.

A report from W. D. Appel, read by Mr. Cady, described continuation of work on calibration of a standard time of lamp exposure for fading tests, and there followed discussion of a name for this standard. Mr. Cady submitted description and photographs of the oven recommended for testing fastness of dyed rayon to gas fading, which were accepted for publication with the test method in the Year Book.

Dr. Meili suggested that a test be developed for the fastness of fabrics to color change in hot pleating. A communication from Lt. Col. Scott suggested that the standard illumination for color matching proposed by the Committee on Color be adopted as a tentative standard of the Association. Major Steadman believed that the background also should be specified, and with this reservation Dr. Scott's recommendation was adopted.

Respectfully submitted,

HAROLD C. CHAPIN, *Secretary*.

LAPEL BUTTONS bearing the seal of the Association may be purchased, by members in good standing, from the Secretary for $1.50 each.

Applicants listed in the UNEMPLOYMENT REGISTER should advise the Secretary of the American Dyestuff Reporter IMMEDIATELY when they obtain employment

The Analysis of Cation-Active SOFTENING AGENTS

DANIEL GELTNER and W. W. RAZIM
Onyx Oil & Chemical Company

UNTIL recent years, no permanent softening agent for textiles was known. Numerous compounds were in use, but all of these were removed by washing and thus, from the consumer point of view, served as little more than temporary disguises of the true hand.

The search for a permanent softening agent brought forth a number of organic nitrogen compounds which were found to impart a soft hand to textiles and moreover, to persist after washing. These compounds are generally either amido-amines, imidazolines, oxazolines or amino-esters, neutralized with an appropriate acid.

After an extensive research, our own laboratory brought forth a series of such compounds. These compounds are said to have the property of imparting a remarkable softness, smoothness, and drape to otherwise harsh, rough and coarse fabrics, and in addition will resist many launderings and dry-cleanings.

It is the purpose of this paper to deal with the general analysis of commercial softening agents of the types mentioned above. These nitrogen compounds are usually dispensed as salts in aqueous dispersions, with or without fillers, and in the form of liquids or pastes. A thorough examination of such a softening agent entails the following procedures:

PRELIMINARY EXAMINATION

A sample of the softening agent is examined to determine whether it is of the cation-active type. The general appearance and odor of the product is noted. The typical commercial sample of this type of softener has a sharp acidic odor, although this may be more or less masked by perfuming agents.

A 1 per cent solution is prepared and its pH determined with the glass electrode. The pH will generally be below 7. Four portions of the 1 per cent solution are removed and treated as follows:

To the first portion is added about one-fifth its volume of N sodium hydroxide. An increase in turbidity is indicative of a cation-active compound. A decrease in turbidity may be due to saponification of dispersed fatty acid.

The second portion is similarly treated with about one-fifth its volume of N hydrochloric or sulfuric acid. An increase in turbidity is indicative of an anion-active compound, while a decrease may be due to the neutralization of an organic nitrogen base.

The third portion is treated with an equal volume of a 1 per cent solution of an anion-active compound (e. g. soap or a sulfated alcohol). An increase in turbidity is indicative of a cation-active compound.

The fourth portion is similarly treated with an equal volume of a 1 per cent solution of a cation-active compound (e. g. a fatty amine acetate). An increase in turbidity is indicative of an anion-active compound.

Since a change in turbidity may not be immediately manifested in the tests given above, it is necessary to let the solutions stand for some time before drawing conclusions. Indeed, it is advisable to let the solutions stand overnight and then note any changes. If these preliminary tests indicate the compound to be cation-active, the following procedure is adopted:

ASH

A sample of the unknown is ignited in a crucible. Usually very little ash will be found. This may be further analyzed by the usual methods if desired.

ALCOHOL EXTRACTION

A 20 g. sample is weighed to the nearest decigram and treated with 100-150 ml. of ethanol at 70° C. After sufficient agitation to bring all the alcohol-soluble constituents into solution, the mixture is filtered through a weighed filter paper. The residue is then carefully washed with three successive 50 ml. portions of alcohol at 70° C.

The cation-active compound should be completely soluble in alcohol. Any residue is likely to be a filler (usually dextrin). The amount of such a residue should be determined by drying the filter paper and its contents to constant weight. Tests may then be run on the residue to identify it and determine the constituents quantitatively.

VOLATILE MATTER

A 10-20 g. sample of the product is weighed to the nearest 20 mg. into a 250 cc. Erlenmeyer flask with a ground glass joint. About 80 cc. of xylene and 3 g. of anhydrous potassium acid sulfate are added, together with a few beads to prevent bumping. A Bidwell and Sterling moisture trap is attached after filling the latter with xylene and wetting the ground joint with the same. A reflux condenser is then fitted to the trap and the mixture refluxed until no more moisture appears to come over. Any adherent drops of moisture are carefully dislodged with a nichrome wire inserted through the condenser top; the refluxing is stopped and the volume of condensed moisture read.

This condensate will usually consist of water with or without volatile acids. Although it is conceivably possible that some organic solvent may be present in the condensate in an appreciable quantity, such an occurrence would be unusual. Nevertheless, a convenient test[1] for

alcohols may be made on a portion of the condensate in the following way:

Two drops of the solution are placed in a small test tube with three drops of carbon disulfide and a few centigrams of solid sodium hydroxide. After shaking the mixture for several minutes, two drops of 1 per cent ammonium molybdate solution are added. When the sodium hydroxide has dissolved, the solution is carefully acidified and shaken with 3 drops of chloroform. If the chloroform layer is colored violet, the presence of an alcohol is indicated.

If an alcohol is indicated, it should be determined and corrected for; otherwise the condensate may be assumed to consist of water and volatile acids. About half of the sample is titrated with 0.1 N sodium hydroxide to both methyl orange and phenolphthalein end points. If the titration reveals an absence of acid, the condensate may be assumed to be water. In such a case, the acid used to neutralize the base is non-volatile and will have to be determined on another sample of the softening agent by the usual methods.

If an acid is found to be present and the phenolphthalein and methyl orange end points coincide, the acid is evidently hydrochloric acid and the quantity present may be calculated from the titration figures. If an acid is indicated, but the phenolphthalein and methyl orange end points do not coincide, then a volatile organic acid is present and hydrochloric acid may or may not be present. In this case the sample is titrated for chlorides by Fajans' method, using dichlorofluorescein as an indicator. The chloride thus determined is calculated as hydrochloric acid and subtracted from the total acid found. The difference is volatile organic acid. This is usually formic acid or acetic acid. Formic acid may be detected by its property of reducing Fehling's solution. Acetic acid may be detected[2] by placing 2 drops of the solution on a spot plate together with 1 drop of 5 per cent lanthanum nitrate solution and 1 drop of .01 N iodine solution. A drop of N ammonia solution is added. A blue to brown color developing around the drop of ammonia in several minutes is indicative of acetate ion.

After qualitatively detecting the volatile acid, it is quantitatively analyzed by the usual methods, and the amount present in the condensate is calculated.

If the amounts of alcohol, organic acid and hydrochloric acid are subtracted from the total amount of condensate, the difference may generally be regarded as water, and consequently the percentage of water in the sample may then be calculated. In any case, it is always advisable to determine the total volatile matter by the usual oven-drying method. A determination by this method will not necessarily yield the same percentage of volatile matter as found with the xylol moisture method since a volatile solvent may be present which would be dissolved in the xylol layer.

HYDROLYSIS

A 75 g. sample is weighed to the nearest decigram into a 300 cc. Florence flask. 25 cc. of distilled water and 75 cc. of concentrated hydrochloric acid are added. The flask is then fitted with an air condenser and heated on a steam bath for 24-48 hours, or until a clear layer of fat separates on the surface.

The fatty acids are now removed by the usual ether extraction procedure, heated to constant weight in an oven at 110° C. and identified by the usual determinations such as a saponification number, iodine number, specific gravity, refractive index, etc. It is also advisable to determine any unsaponifiable fatty matter which may be present. The water layer from the extraction is saved for the next procedure.

ISOLATION AND IDENTIFICATION OF THE AMINE

The water layer from the foregoing procedure is evaporated to a syrup, neutralized to phenolphthalein with a saturated solution of sodium hydroxide, and treated with an excess of several milliliters of the same. The amine thus liberated is extracted in a separatory funnel with three successive 25 cc. portions of n-butanol which are then combined. The water layer is now rejected. The butanol extract is dried in the following way:

A pinch of anhydrous sodium sulfate is added to the extract in the separatory funnel. The funnel is now stoppered and shaken. Large discrete crystals should form, but if an aqueous layer separates, it is tapped off and more sodium sulfate added. This is repeated until large crystals appear. Somewhat larger amounts of sodium sulfate are now added until the salt particles no longer hydrate. If this is done properly, the salt will remain loose, generally with a porous mass of crystals just above the stopcock. The extract is now run out and filtered through a small funnel containing a wisp of cotton, loose enough not to impede the flow of liquid, yet full enough to retain the salt particles. If the stopcock of the separatory funnel is only slightly opened, practically all of the salt will remain in the separatory funnel.

The extract is now transferred to a side neck distilling flask to which is fitted a condenser and the butanol is carefully distilled off until the extract has been concentrated to about 15 or 20 cc. At this point, the butanol distillate should be titrated to methyl red with 0.1 N sulfuric acid. There should be no appreciable alkalinity if the distillation has been carefully performed unless some low-boiling amine, such as monoethanolamine is present. Therefore, any appreciable alkalinity should serve as a clue to the presence of such an amine.

The amine extract is now transferred to a 30 cc. round bottom flask to which is filtered a water cooled condenser, and concentrated further by heating the flask in an oil bath and distilling over all the remaining solvent at an oil bath temperature of 190° C. The distillate is checked for alkalinity as in the previous distillation.

The oil bath is now allowed to cool and the condenser is connected to a suitable receiver which is in turn connected to an aspirator or vacuum pump. The temperature

PROPERTIES OF COMMERCIALLY AVAILABLE AMINES

Name	Van Slyke Kjeldahl	Refractive Index 25°	% N (Theoretical)	Molecular Weight	Sp. G. 20/°20°/C.	Boiling Point	Solubility in Water
Diethylamine	0	1.384	19.2	73.1	0.711	55.5	
Triethylamine	0	1.398	13.85	101.1	0.723	89.5	slight
n-dibutylamine	0	1.415	10.8	129.2	0.7680	159	insolube
Diamylamine	0	1.425	8.9	157.2	0.775	202° (at 745)	slight
n-tributlyamine	0	1.428	7.56	185.2	0.7782	214°	insoluble
Triamylamine	0	1.437	5.90	237.2	0.795	Ca. 250°	insoluble
n-dioctylamine	0	1.438	5.8	241.5			
Methylethanolamine	0	1.441	18.7	75.1			
Di-2-ethylhexylamine	0	1.447	5.8	241.5	0.8062	281.1	insoluble
Morpholine	0	1.451	16.1	87.1	1.0017	128.9	
Dimethylcyclohexylamine	0	1.454	11.0	127			
Diethylcyclohexylamine	0	1.456	9.0	155			
Diethanolamine	0	1.474	13.3	105.1	1.0922	268	
Morpholine-ethanol	0	1.476	10.7	131.1	1.071	225.5	
Triethanolamine	0	1.481	9.4	149.2	1.1258	208 (10 mm.)	
Benzyldiethylamine	0	1.496	8.6	163.1	0.89	210°	
Diethylaminoethanol	0		11.9	117.2	0.8851	162	
Phenylethanolamine	0		10.2	137.2	1.0970	285	5% at 20°
Phenyldiethanolamine	0		7.7	181.2			4% at 20°
Ethylphenylethanolamine	0		8.5	165.2	1.04		insoluble
Tri-isopropanolamine	0		7.3	191.3	0.9996 50°/20°	305.4	
Hydroxyethyltriethylenetetramine	0.25	1.505	29.5	190			
Tetraethylenepentamine	0.4	1.506	37.0	189.3	0.9980	333	
Hydroxyethylenediamine	0.5	1.483	37.2	104.2	1.0304	243.7	
Triethylenetetramine	0.5	1.497	38.3	146.2	0.9818	277.5	
Diethylenetriamine	0.67	1.483	40.7	103.2	0.9542	206.7	
n-butylamine	1.0	1.398	19.2	73.1	0.7285	77.1	
n-amylamine	1.0	1.415	16.1	87.1	0.77	104°	
Isopropylamine	1.0	1.442	23.7	59.1	0.690	Ca. 33°	
Propylenediamine	1.0	1.443	37.8	74.13	0.8732	119.7	
Dodecylamine	1.0	1.446	5.64	248			
2-Amino-1-butanol	1.0	1.446	15.7	89.14	0.944	178	
2-Amino-2-methyl-1-propanol	1.0	1.446	15.7	89.14	0.934	165	
Ethanolamine	1.0	1.452	22.9	61.1	1.0179	170.5	
2-Amino-2-ethyl-1, 3-propanediol	1.0	1.488	11.7	119.2	1.009	152 (10 mm.)	
2-Ethylhexylamine	1.0		10.8	129.2	0.7920	167.5	
Ethylenediamine	1.0		46.7	60.1	0.8995	117.2	
2-Amino-2-methyl-1, 3-propanediol	1.0		13.3	105.1		152 (10 mm.)	
Tris (hydroxymethyl) aminomethane	1.0		11.6	121.1		220 (10 mm.)	

Note: The refractive indices given above have been determined on commercial samples rather than on pure compounds.

is gradually raised and the amine distilled under vacuum, bringing the oil bath temperature as high as 280° C., if necessary. If the amine appears to be a mixture, a fractionation should be performed under reduced pressure; this will usually require suitable semi-micro apparatus. Indeed, all the apparatus described may be replaced with proportionally smaller apparatus, if it is desired to run the analysis on a smaller sample. The amounts of reagents must, of course, be proportionally reduced.

Having isolated the amine or amines, a semi-micro Kjeldahl determination is made on 50 mg. samples. The apparatus used at this laboratory is the one described by Redemann[3]. The absorption of ammonia is carried out by running the ammonia into 30 ml. of saturated boric acid solution. The ammonia may then be titrated directly with 0.1 N hydrochloric or sulfuric acid, using a mixture of methyl red and methylene blue as the indicator. If properly mixed, this indicator will give a practically colorless end point; distinctly green on the basic side and purplish on the acid side.

A micro Van Slyke determination is also run on the amine or amines. The per cent nitrogen found by the Van Slyke method is divided by the per cent nitrogen given by the Kjeldahl method. The ratio so obtained is valuable in that it represents the proportion of nitrogen which is present as primary amino nitrogen. The table shown at the top of this page classifies all the principal commercially available amines according to this ratio. The members of each group are arranged according to their refractive index. Knowing the per cent total nitrogen (Kjeldahl) and the refractive index (which should be determined), the amine is identified by comparing these figures with those of the amines in the same group, as given in the table. Should there be any doubt, a specific gravity determination and/or a boiling point determination can be made and the results compared with the figures given in the table.

A convenient micro-method for the simultaneous determination of the boiling points of these amines is that described by Emich and Schneider[4] and elaborated upon by Benedetti-Pichler and Spikes[5].

A Kjeldahl analysis should also be made upon the original sample of softener. Knowing the percentage of nitrogen present in the amine and knowing the percentage of nitrogen in the original sample, the percentage of amine in the sample may be calculated.

COMMENTS

It is to be noted that the acid hydrolysis may decompose some amines to form lower amines. These may therefore occur in traces. A small amount of monoethanolamine may

also result from the decomposition of hydroxyethyl ethylenediamine or diethylene triamine.

Since it is not the purpose of this paper to cover every phase of the analysis of cation-active softening agents, many details such as the quantitative analysis of fillers, organic acids, etc., have been omitted. Obviously, any paper purporting to cover all these details would have to be book-length.

ACKNOWLEDGMENTS

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Oxidation of Cellulose—

(Continued from page 400)

tity of glacial acetic acid. The oxycellulose was treated with this solution at 25° C.

Carboxyl contents were determined by measuring the amount of silver bound by the fiber from a solution of silver-*o*-nitrophenolate according to the procedure described by Sookne and Harris[10].

Two types of fluidity measurements were made. The first consisted in measurements on dispersions of cellulose in cuprammonium hydroxide solution by the method of Clibbens and Geake[11] as modified by Mease[12]. The second involves the nitration of the cellulose, as recommended by Berl[13], and the determination of the fluidities of dispersions of the nitrocellulose in acetone. The necessity for making both types of fluidity measurements was made clear in the extensive investigations by Davidson who showed that a measure of the true chain length of certain oxycelluloses containing alkali-sensitive linkages could not be obtained by dissolution of the material in an alkaline solvent such as cuprammonium hydroxide solution. In order to avoid the degradation that occurs when the oxycellulose is dispersed in such alkaline solutions, recourse was had to the nitro-cellulose method.

The nitrocellulose fluidities were calculated from the times of flow of solutions through an Ostwald viscometer at 20° C. To reduce the time required to make measurements, solutions containing 0.1 g. (rather than 0.25 g.) of nitrocellulose per 100 ml. of acetone were used. To render the results comparable to the findings published by others, fluidities were calculated for solutions having a concentration of 0.25 g. per 100 ml, by means of Hess and Philipoff's modification[14] of Baker's equation:

$$\eta_{rel}^{\frac{1}{8}} = 1 + \frac{[\eta]}{8} C$$

in which η_{rel} = relative viscosity
$[\eta]$ = a constant
C = concentration in g/100 ml. solvent

The fluidities were readily calculated from the η_{rel}. The fluidity of the solution having the concentration of 0.25 g. per 100 ml. solvent is referred to as the "nitrocellulose fluidity." The nitrocelluloses derived from samples which had consumed more than 0.05 milliequivalents of oxidant per gram were not completely soluble in acetone[15]. The nitrocellulose fluidity of the soluble portion was determined after removing the insoluble material by centrifuging and making the necessary corrections for concentration. It is recognized that the solution thus obtained is not representative of the entire material. It is noteworthy that when the aldehyde groups are oxidized to carboxyl groups, the derived nitrocelluloses are completely soluble.

The sensitivity of periodic acid-oxycellulose to alkalies is indicated by the increase in nitrocellulose fluidity that occurs when such materials are treated with dilute sodium hydroxide[15]. Preliminary experiments showed that the maximum increase in nitrocellulose fluidity was obtained by a one-hour boil (under reflux) in 0.25 *N* NaOH, the ratio of sample to solution being 1 to 200. The scission of all alkali-labile linkages is completed under these conditions. The "alkali boil" referred to hereafter represents such a treatment, and the loss in weight that occurs during the treatment, expressed in per cent, is hereafter referred to as "alkali solubility." It should be further pointed out that while the nitrocellulose fluidity does reach a maximum and constant value after a one-hour boil in the alkali, the alkali-solubility value rises rapidly during the first hour but continues to rise at a very much slower and diminishing rate thereafter. Even after 8 hours the slow dissolution of the cellulose continues to take place.

III. RESULTS AND DISCUSSION

1. Estimation of Oxidant Consumed by Cellulose

As indicated in Figure 1, the consumption of periodic

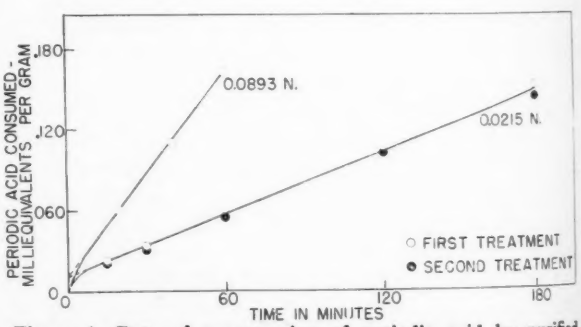

Figure 1—Rate of consumption of periodic acid by purified cotton from solutions at pH 4.6 and 25° C.

acid during early stages of oxidation (that is, oxidation of less than 2 per cent of the glucose residues) is directly proportional to time. It was observed, however, that an

initial rapid disappearance of a small portion of the oxidant always occurs. This could conceivably be due either to the oxidation of some rapidly oxidizable material, or to some unexplained adsorption of the oxidant by the fibers. That the latter is the case was indicated by the following experiment. A sample of purified cotton was subjected to two consecutive oxidations with a 0.0215 *N* solution of periodic acid at pH 4.6. The results shown in the lower curve in Figure 1, indicate the absence of an easily oxidizable substance, since the same initial rapid disappearance of periodic acid was observed on the second treatment. As also indicated in the figure, a fourfold increase in the concentration of periodic acid did not appreciably alter the amount of oxidant initially taken up. Extrapolation of the straight-line portion of the curves to zero time indicated that 0.012 milliequivalents of periodic acid per gram of cellulose have apparently been adsorbed by the fibers in this manner, and accordingly this amount is deducted from the total apparent amount of oxidant consumed in order to obtain a measure of the periodic acid which has been utilized in the oxidation of the cellulose.

The adsorption noted is not unique with periodic acid, inasmuch as similar adsorptions by cellulose of other oxidizing agents such as hypochlorite[16], chromic acid[17], and iodine[1] have been reported in the literature.

2. The Course of Oxidation of Cellulose by Periodic Acid

It would be expected that complete oxidation of cellulose by periodic acid, according to equation 1, would require one atom of oxygen per glucose unit. However, as shown by Jackson and Hudson[6], the actual consumption of oxidant considerably exceeds this theoretical amount, a fact which led these investigators to the conclusion that secondary reactions undoubtedly occurred during prolonged contact of the cellulose with the oxidant. This conclusion was supported by the work of Davidson[15] who found that on extensive oxidation, appreciable quantities of formaldehyde, formic acid and carbon dioxide were formed.

On the other hand, it appears that during the early stages of oxidative attack, the reaction does proceed principally according to equation 1. The data in Table 2

TABLE 2
Aldehyde content of periodic acid-oxycelluloses

Net oxidant consumed	*Aldehyde content*	*Percent of total glucose units oxidized, calculated from aldehyde content*
m-eq/g	*mmol/g*	
0.043	0.041	0.33
.064	.069	.56
.081	.081	.66
.095	.097	.79
.115	.113	.92
.153	.156	1.3

clearly show that in this range of oxidation, one aldehyde group is produced for each equivalent of periodic acid consumer; i.e., two aldehyde groups per mole of periodic acid.

The aldehyde groups in oxycellulose were determined by measuring the iodine consumed during the quantitative oxidation of aldehyde groups to carboxyl groups, according to the modified procedure described in Section II, 2. The method is similar to that used in the estimation of aldehyde groups in hydrocellulose[1] and it was expected that, similarly, an analysis for carboxyl groups in the fiber after treatment with iodine would also demonstrate that the consumption of iodine was due to reaction with aldehyde groups. Unfortunately, when the oxycellulose was placed in the mildly alkaline buffer used in the iodine titration, reducing groups were dissolved, so that the carboxyl groups left in the fiber could not be equivalent to the reducing groups originally present. However, a check of the oxidation reaction was made by the method described below.

The recent work of Jeanes and Isbell[18], showing that aldoses are readily oxidized to the corresponding aldonic acids by treatment with chlorous acid at pH 2.2 provided a means for oxidation in acid solution. Thus, treatment of the samples for 40 to 60 minutes with chlorous acid resulted in quantitative oxidation of the aldehyde groups to carboxyl groups as shown in Table 3. Furthermore

TABLE 3
Aldehyde and carboxyl contents of periodic acid-oxycellulose after chlorous acid treatment

I Initial aldehyde content	*II Aldehyde content after $HClO_2$*	*III Carboxyl content after $HClO_2$*	*Sum of columns II & III*
mmols/g	*mmols/g*	*mmols/g*	*mmols/g*
0.041	0.0000	0.037	0.037
.095	.000	.097	.097
.156	.010	.141	.151
.041	.011	.026	.037
.095	.012	.086	.098
.156	.022	.125	.147

the estimation of the latter affords an independent check on the aldehyde content obtained by the iodine method.

In samples where the oxidation of the aldehyde groups was not completed, it may be seen that the sums of the carboxyl and aldehyde contents after the chlorous acid treatments agree within the experimental error with the aldehyde contents of the periodic acid-oxycelluloses found by the iodine method of analysis. This may be taken to indicate that no new reducing or acidic groups are formed in the oxycellulose as a result of the chlorous acid treatment. Finally, it was found that a similar cholorous acid treatment had no measurable effect on purified cotton cellulose.

3. Relation Between Aldehyde Content and Alkali Sensitivity

As already noted by Davidson, the most characteristic property of the periodic acid-oxycelluloses is the ease with which they are degraded by alkaline solutions. This instability is explained by the fact that rupture of a glucose residue according to equation 1 results in the formation of alkali-sensitive linkages which undergo a scission in alkaline solutions. Thus, Davidson was able to show that periodic acid-oxycelluloses exhibited low nitrocellulose

fluidities (long chains) and high cuprammonium fluidities (short chains). When the samples were subjected to an alkali boil, the nitrocellulose fluidities were considerably increased but the cuprammonium fluidity remained unchanged. In other words, the treatment with alkali resulted in scission of all of the alkali-labile linkages, a process that is also accomplished in the highly alkaline cuprammonium solution.

Further support for this explanation was obtained in the present investigation. As shown in Figure 2, there is

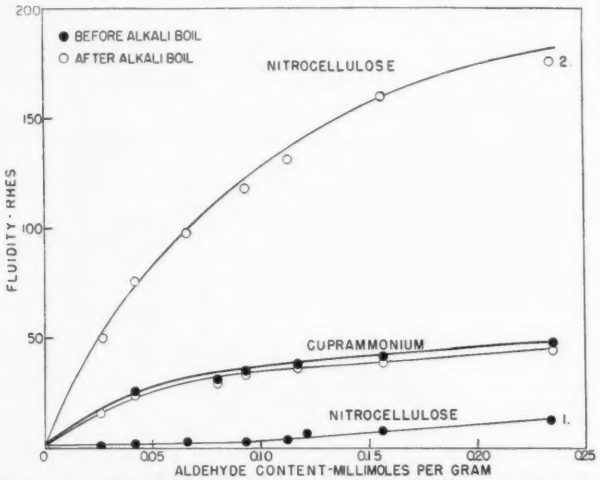

Figure 2—Fluidities of periodic acid-oxycelluloses, oxidized to various alhehyde contents, before and after boiling in 0.25 N NaOH for one hour.

only a slight increase in nitrocellulose fluidity with increase in aldehyde content (curve 1) of a series of oxycelluloses, indicating that although an appreciable number of glucose rings had been ruptured, the molecular chain length had not been correspondingly shortened. That the chains contained alkali-labile linkages, however, is suggested by the high nitrocellulose fluidities of a similar set of samples after the alkali boil treatment (curve 2). In contrast, the cuprammonium fluidities (figure 2) increased rapidly with increasing aldehyde content, regardless of whether the samples were subjected to preliminary alkali treatment, indicating as pointed out earlier, that scission of all alkali-labile linkages in this type of oxycellulose is completed during dissolution of the oxycellulose in cuprammonium solution.

The question arises as to whether the sensitivity to alkalies of this type of oxycellulose depends on the rupture of the glucose ring between carbon atoms 2 and 3 or on a specific instability towards alkali of the dialdehyde formed in the oxidation process. In order to throw some light on this question, samples of oxycelluloses were treated with chlorous acid in order to oxidize the aldehyde groups to carboxyl groups.

As shown in figure 3, the effect of this treatment in every case was to lower the alkali-solubility to nearly zero. If the alkali-solubility values represented a true solubility phenomenon, it might have been expected that conversion

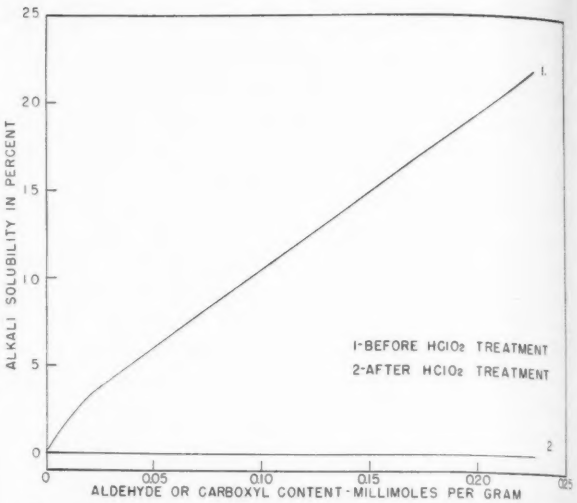

Figure 3—Percent weight loss caused by treatment of oxycelluloses with boiling 0.25 N NaOH for one hour:
1. Periodic acid-oxycellulose; percent weight loss plotted against aldehyde content.
2. Periodic acid-oxycellulose oxidized further with chlorous acid; percent weight loss plotted against carboxyl content.

of aldehyde to carboxyl groups would have resulted in an increase in solubility. Since this is not the case, it is concluded that the relatively high alkali-solubilities of the original oxycelluloses are related to a specific instability, involving the presence of aldehyde groups, of the products toward alkali. Further support for this conclusion is found in Figure 4. Whereas the nitrocellulose fluidities

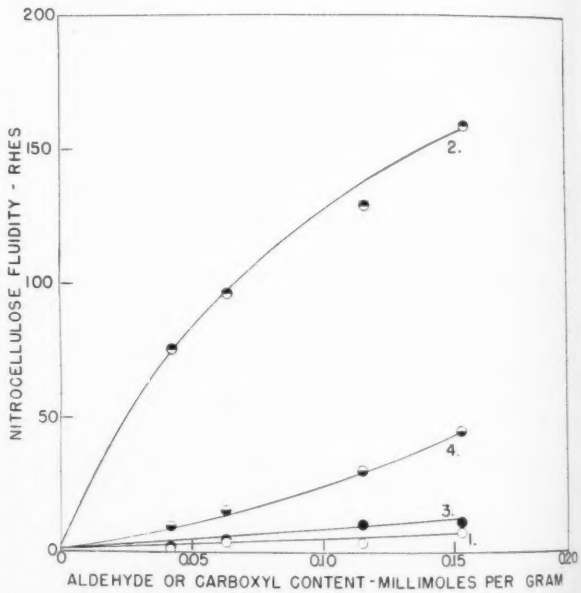

Figure 4—Nitrocellulose fluidities of periodic acid oxycelluloses
Curve 1. Untreated oxycellulose; fluidity plotted against aldehyde content.
Curve 2. Oxycellulose after alkali boil; fluidity plotted against aldehyde content before boil.
Curve 3—Oxycellulose after chlorous acid treatment; fluidity plotted against carboxyl content.
Curve 4. Chlorous acid-treated oxycellulose after alkali boil; fluidity plotted against carboxyl content before boil.

of the oxycelluloses are greatly increased by an alkali boil, treatment of the oxycellulose with chlorous acid yielded products whose nitrocellulose fluidity was much less affected. The results, leave little doubt that the sensitivity to alkali of these oxycelluloses does not depend solely on the rupture of the glucose ring between carbon atoms 2 and 3, but is related to the specific instability towards alkali of the oxidation product formed according to equation 1.

The authors are indebted to A. Eleanor Davis for assistance in making a number of the determinations reported in this paper.

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TRADE NOTES • NEW PRODUCTS

OBITUARY

FRANK S. DUNHAM

FRANK S. DUNHAM, who had been associated with the Permutit Company in the sale of water conditioning equipment for approximately 25 years, died in Chicago on July 23rd.

Mr. Dunham was born in Baltimore on March 8, 1876. He began engineering studies at the Armour Institute of Technology and completed his work in chemical engineering at the University of Chicago. Following graduation, he entered the employ of the Hydrox Corporation of Chicago and subsequently was a chemical engineer with the Kennicott Company, manufacturers of water softening equipment. He then became associated with the Permutit Company with whom he remained until his retirement in 1940.

He served as a captain in the Illinois National Guard and was a veteran of the Spanish-American war.

He is survived by his wife and two sisters.

• GENERAL RELEASE

General Dyestuff Corp., 435 Hudson Street, New York City, has recently released a circular describing the following product:—

Acid Chrome Blue 3 G N A—a new addition to the General line of chrome colors. When applied by either the afterchrome or the silk-white method, it produces greenish shades of blue said to be of very good fastness to light, fulling, washing, perspiration, water, salt-water, crocking, hot pressing and alkali. It is stated that it will be found especially valuable for dyeing woolen piece goods by the silk-white method when silk decorations are to be left unstained. Circular G-309.

• PERMALON

Permalon, manufactured by The Hart Products Corporation, of 1440 Broadway, New York City, is a new permanent cellulose finish available in several viscosities for many types of knitted and woven fabrics.

Mosquito netting (marquisette, tricot, bobinette and lace) processed with Permalon, is said to meet Government specifications. It is stated that it has been tested and approved by the Government supply departments. It is claimed that millions of yards of mosquito netting finished with Permalon are already in use by the armed forces.

Among the advantages claimed are the following:

Very Low Shrinkage—well below Government requirements; Even Penetration —produces a clean, smooth surface; Eliminates Slippage—permanent adhesion of warp and filling yarns; Spreads Evenly With Pigment Colors — uniform, clear shades; Economical—low cost helps meet Government price standards; Quick Delivery—ample stocks on hand to meet all requirements.

The manufacturer will be glad to arrange for a demonstration and send a member of their technical-service staff to any interested mill.

• JOINS HOOKER

Ralph E. Davis, Jr., has joined the Sales Department of the Hooker Electrochemical Company, Niagara Falls, N. Y. Mr. Davis has been superintendent of the tannery of Maylender Bros., glove and garment leather manufacturers of Johnstown, N. Y., for the past six years and previously was superintendent of the tannery of Martin Littell Leather Corporation of Gloversville, N. Y. The Hooker Electrochemical Company manufactures caustic soda, liquid chlorine, chlorinated paraffin, benzoate of soda, benzoic acid, monochlorbenzene, sodium sulfide, sodium sulhydrate, thionyl chloride and about 70 other products.

• LABEL PLAN FOR DYED FABRICS

The Federal Trade Commission on August 5th issued proposals for setting up a system of grade labeling on all dyed fabrics. The regulations will be debated at an open hearing on August 18th before they become compulsory.

They specify that such terms as "tubfast," "colorfast," "washproof," or "vat dyes" cannot be used, and set up grade standards for fabric fastness to light, washing, dry cleaning, pressing, perspiration, crocking and gas. In each category, prescribed tests must be passed before the dyed fabric can be graded as A, B, C or D.

Under the proposed regulations, however, it is not mandatory that labels carrying these grades be attached to all products on sale at retail. The force of the order would lie in the fact that no salesman could claim any degree of color fastness unless the claim were backed by the specified grade tests.

The issuance of the proposed regulations won the immediate approval of the consumer division of the Office of Price Administration, the American Association of Home Economics and the American Association of University Women.

Among the descriptive terms ruled out under the proposed regulations are:

Sunfast, fast to light, light fast, guaranteed fast colors, fast color, color fast, will not fade, fade proof, tub fast, washproof, wash fast, colors fast to washing, fast prints, fast, fast dye, fast to dry

cleaning, fast to perspiration, fast to rubbing, fast to pressing, unfadable, or any similar terms "when the textile does not have such sufficient color-fastness as will prevent any and all loss or change of color." The terms "washable," "launderable," and "tubbable" may not be used unless the textile meets grade A specifications. References to vat dyes may not be used unless the "true color fastness" is also given in terms of grades.

• TESTIMONIAL DINNER

Milton Kutz, who started work as an office boy 45 years ago on August 4 and now is assistant to the general manager of the Du Pont Company's Electrochemicals Department, was the guest of honor at a testimonial dinner by his associates at the Hotel du Pont.

An engraved mahogany mantle clock was presented to Mr. Kutz by Colby Dill, special assistant to the general manager. Congratulatory messages from leaders of the chemical industry were read, and his career was reviewed in speeches by his associates.

Mr. Kutz was born in Logansport, Ind., on October 26, 1883, and was educated in the public schools of that town. At the age of 13, on August 4, 1897, he started to work for The Roessler & Hasslacher Company as an office boy.

He advanced successively to stock clerk, the bookkeeping department, the purchasing department, sales correspondent, salesman, and manager of the Philadelphia office. He was made general sales manager in 1922, a member of the board of directors in 1926, and vice-president and manager of sales in 1928.

When The Roessler & Hasslacher Co. was merged with du Pont in 1930, he was named director of sales. He was made assistant general manager when the R. & H. Chemicals Department, now the Electrochemicals Department, was established in 1933. He relinquished this post this year because of illness, and now serves as sales advisor and assistant to the general manager.

He is a member of the American Chemical Society, the Chemists Club of New York, the Du Pont Country Club, the Concord Country Club and the Philmont Country Club of New York. He is president of The Jewish Federation of Delaware.

• CELLUDYE COLOR CONCENTRATES

The Hart Products Corporation of 1440 Broadway, New York City, announces that it has been appointed exclusive sales agent for Celludye Pigment Color Concentrates for the textile industry.

These colors are manufactured by the Celludye Company, 1270 Sixth Avenue, New York City. They are said to be noted for their fastness, quick penetration and high pigment value. Their use is said to eliminate completely the need for any additional dyeing processes. It is stated that required shades are matched, eliminating the necessity for color blending. It is claimed that its high concentration and low price make it very economical to use.

These colors are said to have proven particularly useful in combination with Permalon, a new permanent cellulose finish, manufactured by The Hart Products Corporation for mosquito netting and other knitted and woven fabrics.

• CALCO SALES REPRESENTATIVE

Robert N. Griswold, a member of the Chicago sales staff of Calco Chemical Division, American Cyanamid Company, has been appointed sales representative in the Miami Valley where he will handle the paper trade.

Mr. Griswold joined the Calco organization in 1935 as a technical representative, coming to Chicago from the Morgan Dyeing & Bleaching Company, Rochelle, Ill. He has a degree of B.S. in Chemical Engineering received at the University of Wisconsin, where he graduated in 1932.

• CLASSIFIED ADVERTISEMENTS •

The rate for "Position Wanted" advertisements in this column is 2 cents a word—with a minimum of 50 cents per insertion. For all other types of advertisements—i.e., help wanted, machinery or supplies for sale—the rate is $6.25 per column inch or less per insertion.

POSITION WANTED: Finisher for all types of woolen and worsted fabrics. 19 years' experience in leading mills. Capable of handling large production. Willing to go anywhere in United States. A-1 references. Write Box No. 387, American Dyestuff Reporter, 440 Fourth Ave., New York, N. Y.

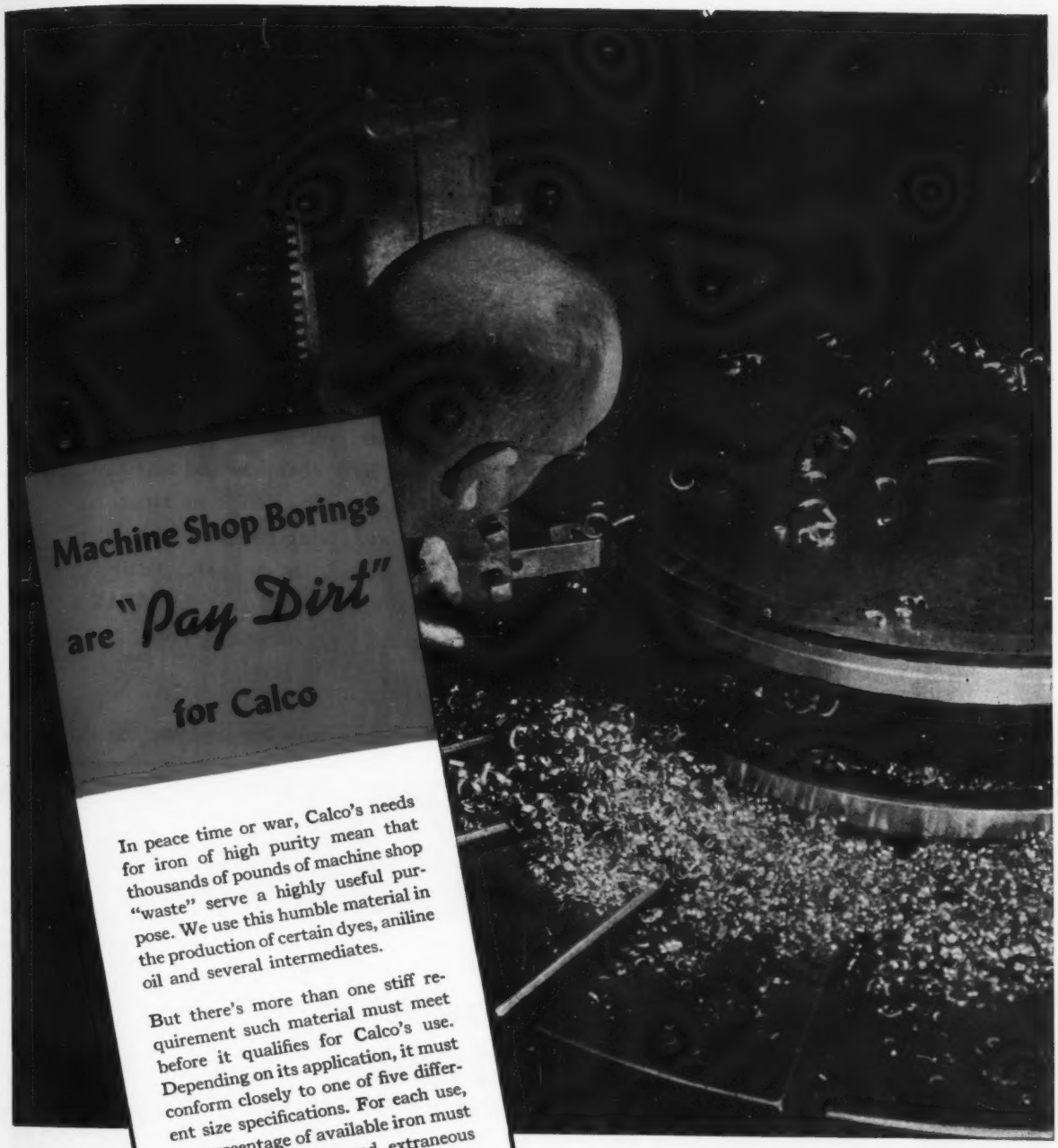
POSITION WANTED: Plant Manager: Experienced plant and production manager in the dyeing, printing and finishing field seeks new connection with progressive organization as plant manager or superintendent of production. Will consider connection, with an organization supplying the trade, in the capacity of representative and demonstrator of their products. At present employed. Write Box No. 389, American Dyestuff Reporter, 440 Fourth Ave., New York, N. Y.

WANTED: Assistant chemist for general laboratory in large southern mill. Should be capable of matching with, and testing all types of dyes, general chemical analysis, and physical testing of yarns and piece goods. State experience, salary expected and draft status. Write Box No. 391, American Dyestuff Reporter, 440 Fourth Ave., New York, N. Y

WANTED: Demonstrator for synthetic resin products for textiles. Applicant must have a good technical background, plus some years of practical experience in the finishing of textiles—principally cotton and rayon piece goods. Write Box No. 396, American Dyestuff Reporter, 440 Fourth Ave., New York, N. Y.

WANTED: Plant man with chemical training and experience to serve as assistant in charge of production in established southern plant manufacturing wet-processing agents and textile specialties. State age, experience and salary expected. Write Box No. 397, American Dyestuff Reporter, 440 Fourth Ave., New York, N. Y.

POSITION WANTED: Textile Colorist. Capable of making and matching of shades for hosiery industry. Past employment—sixteen years as foreman dyer for second largest hosiery mill in south. Willing to prove my ability as dyer for thirty days at my expense. Experienced in dyeing and finishing of pure silk, rayon, cotton, nylon and mixed fibers. Draft status 3-A. Write Box No. 398, American Dyestuff Reporter, 440 Fourth Ave., New York, N. Y.



Machine Shop Borings are "Pay Dirt" for Calco

In peace time or war, Calco's needs for iron of high purity mean that thousands of pounds of machine shop "waste" serve a highly useful purpose. We use this humble material in the production of certain dyes, aniline oil and several intermediates.

But there's more than one stiff requirement such material must meet before it qualifies for Calco's use. Depending on its application, it must conform closely to one of five different size specifications. For each use, the percentage of available iron must be uniformly high—and extraneous foreign matter in appreciable amounts means prompt rejection.

This naturally means that each lot of this important raw material must receive many and careful laboratory tests before it is used. For the simple reason that the quality of *all* Calco dyes and other products begins with the raw materials from which they are produced.

Calco Dyes are Better Buys

Calco

CALCO CHEMICAL DIVISION
AMERICAN CYANAMID COMPANY

Bound Brook, New Jersey

Boston • Philadelphia • Providence • New York • Charlotte • Chicago

STEP UP PRODUCTION WITH

Kali PRODUCTS

Whether it's airplanes, guns, tanks or textiles, today's demand is for more and better production.

KALI TEXTILE CHEMICALS

speed up production by eliminating troubles in processing synthetic yarns, cotton, wool, and blend yarns.

Waterproofing products, one and two bath methods; also containing anti-mildew.

KALI MANUFACTURING CO.
1410 N. Front Street **Philadelphia, Pa.**

SALES REPRESENTATIVES WANTED

One of our clients, a nationally known sales organization handling a line of special, branded, advertised textile finishes, has openings for resident representatives in New England and the South. Applicants must have chemical and textile mill backgrounds. Reply in strict confidence, giving complete history and experience in detail. Address: ROBERT B. GRADY COMPANY, 45 E. 17th St., New York, N. Y.

Manufacturers of Dyestuffs and Chemical Specialties

We offer
Two new cationic type softeners

TEXAMINE

a pure white permanent softener economical to use due to its complete exhaustion qualities

TEXAMINE K

a permanent softener and desizing agent for processing grey goods in the kier eliminating the customary grey desizing.

May we serve you?

COMMONWEALTH COLOR & CHEMICAL CO.

MAIN OFFICE AND WORKS: Nevins, Butler and Baltic Streets, Brooklyn, N. Y.
Philadelphia • Chicago • Charlotte • Gloversville • Kansas City • Montreal

BECCO

ELECTROLYTIC HYDROGEN PEROXIDE

Keep in touch with

BECCO

HYDROGEN PEROXIDE TECHNICAL SERVICE

for accurate, reliable, and comprehensive information on Peroxide Bleaching of all fibres... by all Methods and Procedures including all systems of Continuous Bleaching.

BECCO SALES CORPORATION

BUFFALO, N. Y.

Branch Offices in Boston, New York City, Philadelphia, Chicago and Charlotte

The

Annual Processing Review Number

will appear

December 7, 1942

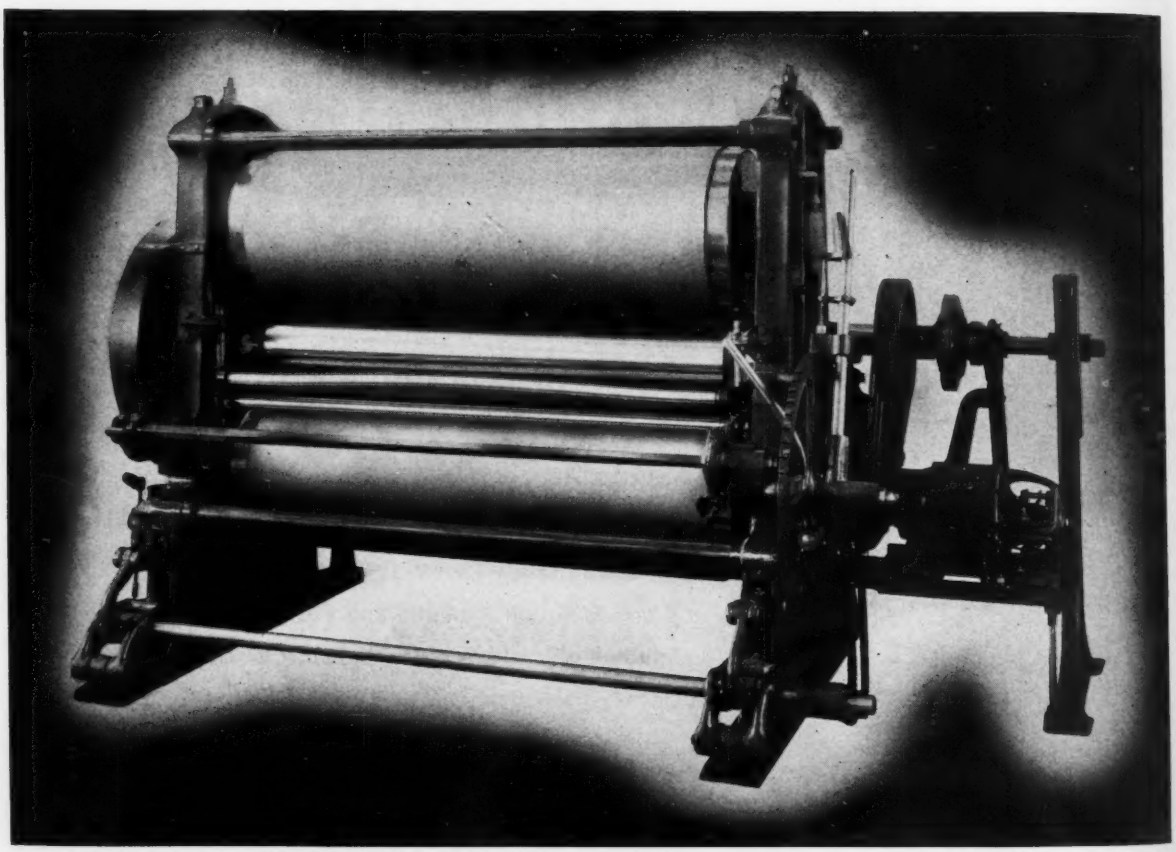
Last advertising forms close

November 23

American

DYESTUFF REPORTER

440 FOURTH AVE. NEW YORK, N. Y.



The V. V. LEVER PRESSURE CALENDER has proved its worth in many plants on a variety of fabrics. Designed for cold or hot calendering at minimum cost. *Constant* pressure is maintained by a system of compound levers with variable weights, and a quadrant ratchet lever for small adjustments. Write for full information and prices.

VAN VLAANDEREN MACHINE COMPANY, 370 Straight St., PATERSON, N. J.

Use them together for

even-running yarn and clearer stitch

Laurel

HYDROCOP and 3B SOFTENER

Two outstanding Laurel Emulsions of timely importance to all who knit or weave cotton. Laurel HYDROCOP and 3B SOFTENER are used to soften and lubricate dyed and bleached yarn. They improve knitting qualities through even- and smooth-running yarn. Restore normal moisture.

Stable, will not turn rancid, nor yellow whites, nor in any way affect subsequent wet processing.

We'll be glad to supply you with a sample and suitable formula to fit your needs.

Send for sample order.

Laurel

SOAP MANUFACTURING CO., Inc.

SOAPS • OILS • FINISHES

Wm. H. Bertolet's Sons — Established 1909
2604 E. Tioga Street — Philadelphia, Pa.
Warehouses: Paterson, N. J. Chattanooga, Tenn Charlotte, N. C

RAPIDASE

THE STANDARD DE-SIZING AGENT

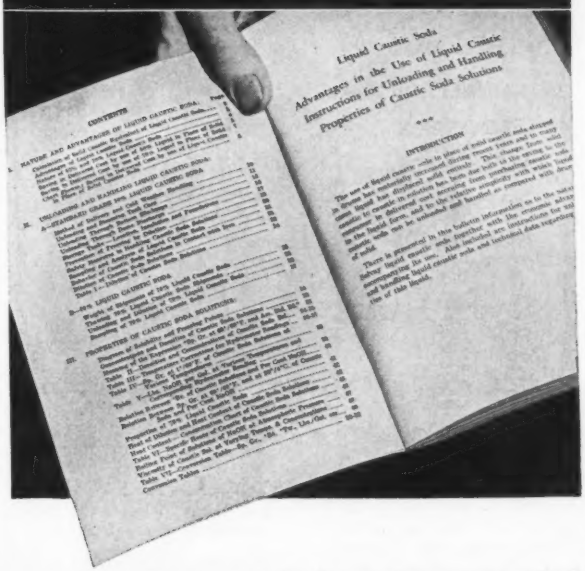
for cottons, rayons and mixed goods

For your de-sizing problems— our technical men are at your service . . . Write or 'phone.

WALLERSTEIN COMPANY, INC.
180 MADISON AVENUE, NEW YORK

BOOK OF THE YEAR

...To The Man Who Needs Down-to-Earth Information On Handling Caustic Soda!



If you are a technician or plant operator who is now "taking over" an assignment which necessitates a more specific knowledge of Caustic Soda than you previously required, *then write today for Solvay Technical Service Bulletin No. 6.*

This bulletin contains time-saving, factual information from which all sales talk has been eliminated. It is full of important tables, charts and other data on the handling of Caustic Soda. It is information which goes straight to the point... helps you improve efficiency, reduce man hours and increase production speed.

Solvay Technical Service Bulletin No. 6 on Liquid Caustic Soda is one of the series of Solvay Bulletins which are constantly being revised on the basis of new findings. For years this technical literature has had the recognition of plant operators, engineers and scholars who realize that such vital operations data concerning alkalies can only originate out of the breadth of accumulated experience Solvay has gained as America's oldest and largest manufacturer of alkalies.

FOR VICTORY BUY UNITED STATES DEFENSE BONDS AND STAMPS

If you don't use Caustic Soda, then send for any of the OTHER SOLVAY BULLETINS listed below, which pertain to your work.

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Alkalies and Chemical Products Manufactured by The Solvay Process Company
40 RECTOR STREET NEW YORK, N. Y.

BRANCH SALES OFFICES:
Boston • Charlotte • Chicago • Cincinnati
Cleveland • Detroit • New Orleans • New York
Philadelphia • Pittsburgh • St. Louis • Syracuse

Use and handling of Caustic Soda • Analysis of Alkalies • Soda Ash Handling • Alkalies and Chlorine in Water Treatment • Analysis of Liquid Chlorine and Bleach • Water Analysis • Calcium Chloride in Refrigeration Road Surface Stabilization • Analysis of Calcium Chloride and Limestone

BURK-SCHIER

WET PROCESSING AGENTS FOR YARNS AND PIECE GOODS

Penetrant 68—Fast wetting agent . . . effective in acid or alkaline baths . . . compatible with all dyestuffs . . . will not scum in hard water.

Mercerizing Assistants—Penetrants for dry mercerizing . . . wetting agents applied directly in mercerizing bath . . . for yarns and piece goods.

Mercerizers Softeners—Various types to meet the individual requirements of yarn and piece goods mercerizers.

Sorbinols—Compounds producing a fabric finish of remarkable absorbency and rewetability . . . goods processed with any of the Burk-Schier Sorbinols are re-wet with great speed and facility.

Diamine Softener—A modified cation-active finishing agent combining a durable finish with exceptional absorbency.

Emulsital W — Emulsified tallow compounded only from choice grade of pure white tallow . . . designed for sizing and finishing quality yarns and piece goods.

Soluble Oils — Self-emulsifying wet-processing agents applicable to a wide range of dyeing and finishing operations.

Tenesol — Yarn conditioning agent . . . sets the twist and assures regain.

Burkol—Synthetic organic detergent . . . effective in acid or alkaline baths.

Kier-Compound — Sodium oleate-pine oil kier assistant.

Creamsol — Concentrated stearic softener . . . ideal for bleach goods . . . imparts soft pleasing handle.

Metasol — A valuable aid in all wet processing operations (send for handbook).

Hy-Pel—An effective, economical water repellent.

Pine Solvent XX — For scouring-bleaching-dyeing. Wets . . . Penetrates . . . Suspends . . . Disperses.

Compounds 743-DS — An organic scrooping agent of proven merit.

Aromine — Wetting agent, dyeing assistant, water normalizer . . . aid to level dyeing and clear bright colors.

Sulphonated Oils — Standard or special grades and finishes.

Burk-Schier Finishes—Cation-active softening and finishing agents . . . Effective . . . Economical . . . Durable.

BURKART-SCHIER CHEMICAL CO.

BURK-SCHIER CHATTANOOGA, TENNESSEE BURK-SCHIER

MANUFACTURING CHEMISTS FOR THE TEXTILE INDUSTRY

• INDEX •
TO ADVERTISERS

Alrose Chemical Co.
Althouse Chemical Co.
Amalgamated Chemical Corp.
American Aniline Products, Inc. III
American Cyanamid & Chemical Corp. Back Cover
American Dyewood Co.
Ansul Chemical Co.
Aqua-Sec Corp.
Aridye Corp.
Arkansas Company, Inc. IV
Arnold-Hoffman & Co., Inc.
Atlas Electric Corp.
Becco Sales Corp. XVII
Bick & Co., Inc.
Blickman, Inc., S.
Burkart-Schier Chemical Co. XX
Butterworth & Sons Co., H. W. Second Cover
Calco Chemical Division, American Cyanamid Co. XV
Campbell & Co., Inc., John
Carbide & Carbon Chemicals Corp.
Carbic Color & Chemical Co., Inc. XII
Ciba Company, Inc.
Colgate-Palmolive-Peet Co.
Commonwealth Color & Chemical Co. XVI
Du Pont de Nemours & Co., Inc., E. I.
Dyestuffs Division Front Cover
Fine Chemicals Division
Electrochemicals Dept.
Emery Industries, Inc.
Exact Weight Scale Co.
Fancourt & Co., W. F.
Gardinol Corp.
Geigy Company, Inc.
General Chemical Co.
General Dyestuff Corp. V
Glyco Products Co., Inc.
Grady Co., Robert B. XVI
Hart Products Corp. IX
Hercules Powder Co.
Naval Stores Department
Synthetics Department
Hotel Manger
Hooker Electrochemical Co.
Houghton & Co., E. F.
Kali Manufacturing Co. XVI
Kelco Co.
Laurel Soap Mfg. Co. XIX
Mathieson Alkali Works, Inc.
National Aniline Division, Allied Chemical & Dye Corp. XIV
National Carbon Co., Inc., Carbon Sales Division VI
National Oil Products Co.
Neville Co., The
Nyanza Color & Chemical Co.
Onyx Oil & Chemical Co., Inc. VII
Perkins & Son, B. F. X
Philadelphia Quartz Co.
Prince George Hotel
Procter & Gamble
Riggs & Lombard, Inc.
Rodney Hunt Machine Co.
Rohm & Haas Co. XXII
Royce Chemical Co.
Sandoz Chemical Works XI
Scholler Bros., Inc. Third Cover
Socony-Vacuum Oil Co., Inc.
Solvay Sales Corp. XIX
Sonneborn Sons, Inc., L. XXI
Stein, Hall & Co., Inc.
Union Carbide & Carbon Corp. VI
Van Vlaanderen Machine Co. XVIII
Virginia Smelting Co.
Wallerstein Co., Inc. XIX
Warner Chemical Co.
Warwick Chemical Co.
Wolf & Co., Jacques
Young Aniline Works
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HERE'S the water repellent finish more and more finishing mills are turning to these days! Textarid CS has been developed specifically for easy, economical application to army tent ducks and all other types of U. S. service fabrics.

Textarid CS (Regular Textarid to which an effective mildew-proofing chemical has been added in an amount substantially exceeding Army requirements) passes with flying colors the standard Army spray and hydrostatic tests as called for by U. S. Army Quartermaster specifications.

Where a mildew-proofed water repellent is not required, Regular Textarid meets all standard requirements for water repellency. Use it, too, on all cotton, rayon, acetate, woolen, worsted, felt and imitation leather fabrics.

TEXTARID
PROCESSED
WATER REPELLENT

Where Quality Counts—Count on Sonneborn

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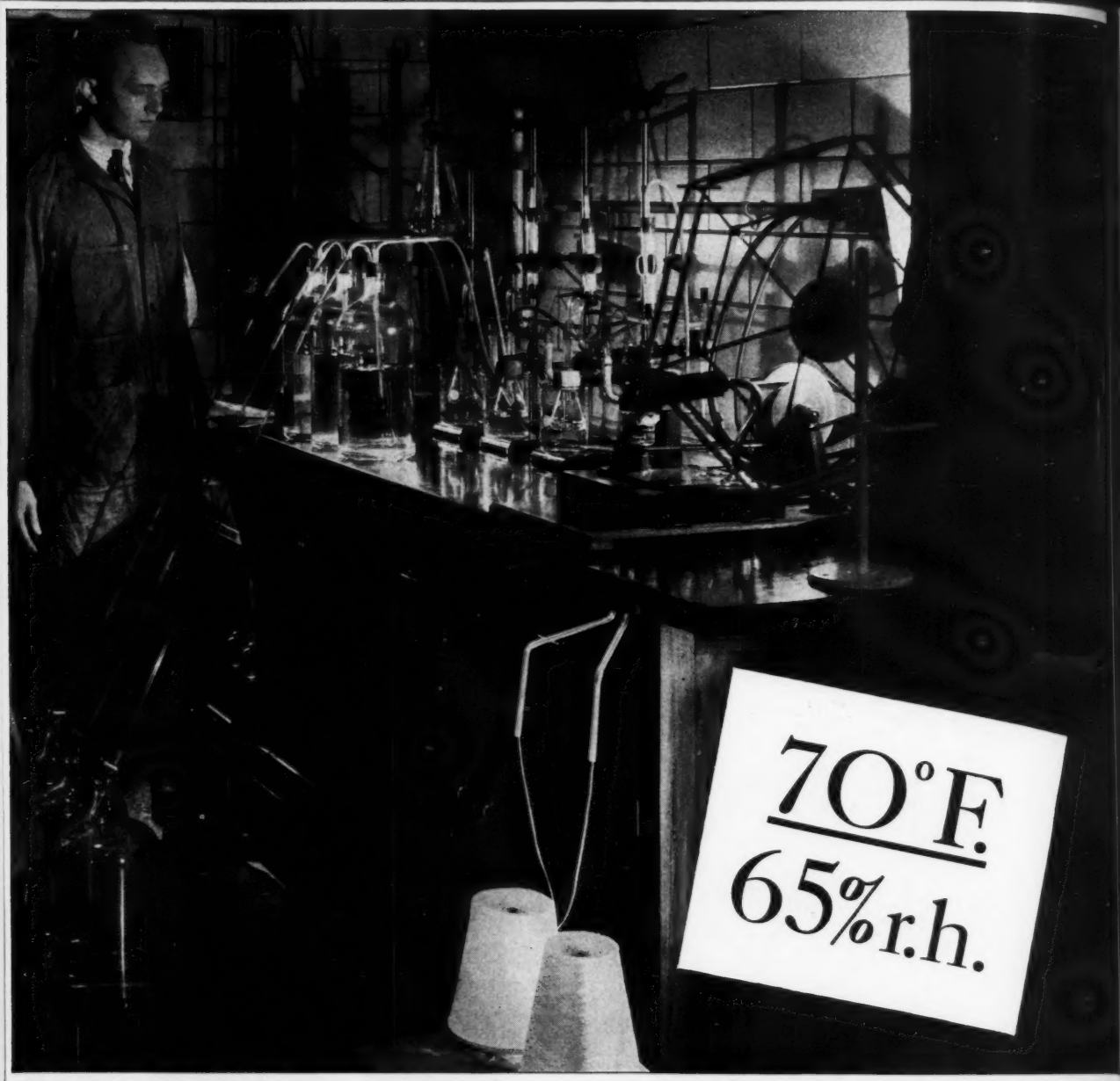
Manufacturing Chemists and Oil Refiners for the Textile Industry

Plant and Laboratories, Nutley, N. J. · Refineries, Petrolia and Franklin, Pa.



EVALUATION LABORATORY — The photograph shows one corner of the Rohm & Haas Textile Evaluation Laboratory where new products are tested, fabric treatments evaluated, and new testing methods developed. In the background is a control board which automatically maintains in the laboratory a constant temperature of 70°F. and a relative humidity of 65%. The highlighted laboratory equipment was designed by the Evaluation Laboratory to determine and evaluate the hygroscopic properties of new resins and sizes. An accurately measured volume of saturated air is passed over the dried samples to determine the moisture pick-up. Other equipment in the photograph includes an abrasion tester, a skein winder, and equipment for the Draves Test.

DON'T GIVE UP UNTIL WE DO! Effective research requires ingenuity! Standard laboratory procedures are not always available and when such is the case, the resourcefulness of the technician determines the effectiveness of the evaluation.

Whenever there is a standard device for testing specific properties, such a machine will almost always be available in the Rohm & Haas Laboratories. In cases when no standard testing device is available, Rohm & Haas research men have developed devices for accomplishing the desired result.

TRITONS – *Synthetic detergents, emulsifying, wetting, and dispersing agents.*

RHOTEXES – *Synthetic gums for sizing and thickening.*

DEGOMMAS – *Concentrated diastatic and proteolytic enzymes for desizing.*

RHOPLEXES – *Aqueous resin dispersions for finishing and coating fabrics.*

RHONITES – *Modified urea formaldehyde resins for textile finishing.*

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WASHINGTON SQUARE, PHILADELPHIA, PA.

Manufacturers of Leather and Textile Specialties and Finishes . . Enzymes . . Crystal-Clear Acrylic Plastics . . Synthetic Insecticides . . Fungicides . . and other Industrial Chemicals

TO HELP MAKE HER WAR-TIME APPAREL BOTH *Attractive & Durable*

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For enchanting Beauty, Allure, Wearability, Spot and Snag resistance, the name Dura Beau* is (and has been for many years) synonymous with countless brands of quality fabrics.

Ask a Scholler Specialist to call.

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for Government Requirements

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ACETATE OF CHROME

BLACK IRON LIQUOR

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